METALLURGIA

The British Journal of Metals

PRINCIPAL CONTENTS IN THIS ISSUE :

The articles in this Journal are indexed in *The Industrial Arts Index*

The Pressed Metal Industry

Productivity Team's Report

MUCH has been done in this country to redress the balance of payments by substantial cuts in imports and in stepping up production for export, much still remains to be done, however, before our impoverished national fortunes can be sufficiently restored to off-set the effects of political and industrial unrest. Although careful adjustments of imports in an effort to balance with exports is commendable the method can only be regarded as of temporary character since imports are vital to the building up of overseas markets for our products : what this country must rely upon to restore equilibrium is greatly increased productivity.

When the Economic Co-operation Act was passed by the United States Congress about two years ago, and the Marshall Plan was put into effect, this question of greater productivity was discussed by Sir Stafford Cripps and Mr. Paul Hoffman from which arose that activity of the Plan known as " Technical Assistance," part of which involves the work of American experts in Europe, but probably the most fruitful part is the contact with United States industry by groups of European industrialists, in which industrial teams from this country have participated. The first group, in the summer of last year, was from the British steel foundry industry. Several groups from other sections of industry have since visited corresponding works in the United States and have issued productivity reports, the more recent of these being the 'Report* of the productivity team from the pressed metal industry, in which recommendations are made for increasing productivity in this country.

The branch of industry represented by this team is mainly occupied with the supply of pressings of various kinds to the motor manufacturing industry. It makes complete all-steel motor car bodies and supplies large quantities of pressings and assemblies for commercial and public service vehicles. The industry also manufactures for the domestic appliance industries, refrigerator cabinets, washing machine cabinets and tubs, steel sinks, panels and assemblies for gas and electric cookers. The largest presses exert a pressure of 1,000 tons, but those making motor panels usually operate at a pressure of 250 and 500 tons. The team, under the leadership of Mr. J. M. Phillips, Managing Director, Motor Panels (Coventry), Ltd., comprised men drawn in equal numbers from the supervisory, technical and workshop levels.

The team appreciates that the large mass production plants of America are able to organise their machines and equipment to a degree which the much lower production rates would not allow in this country. With this point always in mind, the Report deals mainly with

* Published by the Anglo-American Council on Productivity, 21, Tothill Street, London, S.W.1, price 2s. 6d.

the more " non-permanent " types of equipment and methods which could be applied in the average British pressing plant. It is also emphasised that the larger consumer market in the United States gives American manufacturing units much greater scope for quantity production than is afforded to British manufacturers and for this reason the approach to production in that country differs fundamentally from the methods so frequently adopted in Britain.

There were no specific terms of reference ; the broad objects of the team were to study American production methods, techniques and working conditions, with particular reference to their effect on productivity per man hour. Care must be taken to distinguish between increased production and increased productivity ; the former can be obtained by increasing the number of workers and machines, but the latter can only be achieved by keen planning and better utilisation of labour and machines, with a consequent greater output per man and per machine hour. Higher productivity at lower costs is imperative in Britain if a better standard of living is to be achieved. Increased wages not allied to increased productivity can only result in rising prices, reduced demand, reduced production, less wages—a descending spiral.

The American plants visited, and there were many over a wide area, were found to be much more up-to-date in their equipment. The rate of obsolescence was much faster than in British plants owing, in part, to the American industrialists' insistence on having the latest equipment. The American will not use a machine if he can buy another which will give him a cheaper product. American plant does not receive the careful maintenance given to some British equipment ; it is worked to full capacity for a short life and then replaced. A far greater degree of mechanical handling is applicable to America's larger scale of production. In some instances, where tools are set up for long runs in large presses, fully mechanical hands are fitted to the presses, and the saving of power is considerable. The Report describes and illustrates the operation of these mechanical hands and suggests that their use in British press plants will be very limited. In the lower rate production shops in America, however, semi-mechanical handling devices are used extensively ; the rule being that if a mechanical or semi-mechanical device can be used to replace some physical effort a determined effort is made to obtain it.

The Report describes the layout and mass production techniques of American plants and also the methods of material handling. In all the plants visited the greatest emphasis is placed on keeping materials in motion from one operation to another with the minimum of physical effort. It is realised that the flow of materials is the blood stream of business. Manual handling is only permitted when mechanical handling is uneconomical or impossible. The fullest use is made of conveyors of all

types—overhead, fixed belt, roller, gravity chutes—and a wide range of specialised equipment. In certain cases observed, the time between the arrival of the material at one end of the shop to the loading of the finished component in a railway wagon at the other end of the shop was under 15 minutes. The method obviously eliminates the necessity for inter-operational stores, and this was the case in the majority of plants visited. Faster movement of materials with less effort inevitably means a faster production at lower cost, and it is this particular technique, more than any other, which makes American productivity greater than in Britain.

Emphasis is given to the close liaison which exists between the product designer and the tool designer. These skilled men are brought into the earliest discussions with the component designer and the design is made with close watch on cost of manufacture and practicability of manufacture. Particular attention is given to the functional parts of the component and parts which are unseen or are of less importance are given less attention. The result is a saving in tool and production costs. For tooling, jigging and equipment there is intensive pre-production planning. Greatest attention is given to the reduction of physical effort and, where possible, to the elimination of any skilled operation.

With regard to capital equipment the American approach differs from the British approach. The American manufacturer is constantly striving to improve the performance of his machines and as soon as a higher efficiency machine is developed the press users will discard the older equipment. There is a refreshing and positive attitude towards keeping shops equipped with the most up-to-date plant and less emphasis is put on maintenance than in Britain. The Report states that the American does not look upon his machines as a long term investment but rather as something to use to the utmost capacity during the period of peak efficiency. In the large press plants visited a complete range of presses is in use; in the smaller jobbing and batch production plants there is a high degree of specialisation and the range of products is smaller than in corresponding British plants. Specialisation in product and plant leads to a high degree of efficiency and greater productivity.

It is noteworthy that, in the plants visited by the team, there was no evidence of any trade union restrictive practices which limited earnings. On the side of management when piecework prices have been set there is no restriction on earnings. Nor do the unions apply any restrictions to the up-grading of workers. If an unskilled operator shows an aptitude for his work he can be given a semi-skilled job. The Team found instances of semi-skilled men who were being trained for tool making. After a period of training they were paid the full rates. The practice of most of the American firms visited is to engage completely unskilled workers and teach them one single production operation. As they become adept in that job they may be up-graded.

The Report contains eleven illustrated pages describing production equipment and methods and as many pages, also illustrated, deal with tool design and manufacture. A section on labour conditions states that supervision of plants in America is on a high level; the supervisor really functions as management at shop floor level; his job is management and he is not encumbered with clerical work.

As a nation we are consuming more than we produce and, unless we are prepared to sacrifice our standard of living, increased productivity is the only means of restoring equilibrium. The fact that output per man hour in the pressed metal industry is greater than in Britain is generally recognised and the main purpose of this and other visits by experts to inspect American industries is to assist the drive for higher productivity in this country. With managements and factory supervision in America there is a continual striving to obtain optimum machine efficiency, to utilise labour to the best advantage and to continue to improve manufacturing methods and it is this productivity consciousness that is in so much need in Britain.

Among the many recommendations of the Team which are published in this Report is one which emphasises the urgent need for an association of firms in the British pressed metal industry for the interchange of ideas and for research into problems affecting pressing techniques and production methods. We heartily agree and this would seem to offer scope for the expansion of the existing Sheet and Strip Metal Users' Technical Association.

There is much in this Report that is not limited to those engaged in the pressed metal industry, but the most effective return for the efforts of the Team would be the achievement of increased productivity in Britain and all can contribute to this end.

Graduate Courses at Birmingham University

GRADUATE courses in mechanical engineering, chemical engineering and metallurgy will be given in the Faculty of Science, University of Birmingham, during the session 1950–51, which begins on October 2nd. The purpose of the graduate in metallurgy, at the Department of Industrial Metallurgy, is to provide training at a postgraduate level for men who already hold degrees in metallurgy, physics, chemistry or engineering, and who are occupying positions of responsibility in metallurgical industry. In general, it is expected that entrants will have spent some time—e.g., two years or more, in metallurgical industry, but in suitable instances a proportion of metallurgical graduates with less industrial experience may be admitted. In all cases it will be assumed that students possess a knowledge of metallurgy to at least ordinary degree standard.

The course comprises lectures and practical work and is planned to be completed in one academic year. The Diploma in Graduate Studies (Metallurgy) is awarded on satisfactory completion of the course. Most of the practical work is in the form of exercises and investigations in the University laboratories, but provision is also made for some time to be spent in practical studies in industrial concerns.

Lecture courses occupying a total period of eight hours per week are given during normal term time. Men recently graduated at this University in the Departments of Industrial Metallurgy or Theoretical and Physical Metallurgy may only find it necessary to take a portion of the lectures, since some of these will be devoted to revision for the benefit of men separated from their undergraduate period by several years.

The fee for each course will be £70, including lecture, laboratory, examination and membership fees. Applications should be addressed to the Registrar, The University, Edgbaston, Birmingham, 15.

High Temperature Alloys Based on Titanium Carbide

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In the search for materials capable of withstanding severe high temperature operating conditions, many different types of material have been considered. The "hot strength" of cemented carbides is utilised in metal cutting operations and investigations of the properties of alloys, based on titanium carbide and containing chromium and nickel or cobalt, have shown them to possess many of the properties desirable in gas turbine materials. The low specific gravity of alloys containing a large proportion of titanium carbide make them particularly suitable for turbine rotor blades.

CEMENTED carbide cutting tools give superior performance in high speed cutting partly because they have greater resistance to deformation at the high temperatures attained near the cutting edge, than have other tool materials. Reports have been published giving results of hardness tests on cemented carbides at high temperatures, showing a relatively small drop in hardness at temperatures up to 750° C.[1] No other information was available relating to high temperature strength properties of these alloys when this research was started in 1944. It was reasonable to suppose that the carbides themselves, such as WC and TiC, having very high melting points, would have high creep strength and it seemed quite probable that in alloys such as cemented carbides, which contain a high proportion of carbide in their structure, the carbide would play a large part in determining the properties.

The normal cutting tool materials, however, all contain a large proportion of tungsten carbide (WC). They oxidise extremely rapidly when heated in air to temperatures over 600° C. and have the added disadvantage of very high specific gravity (15·67 for WC). Titanium carbide (TiC) is used together with tungsten carbide in many grades of tool materials, and experiments soon showed that alloys of titanium carbide, bonded with nickel or cobalt, have much better oxidation resistance than alloys rich in tungsten carbide. Further improvement was obtained by the addition of a small proportion of chromium. Titanium carbide has the added advantage of a low specific gravity, 4·91.

Alloys of a range of compositions, containing titanium carbide, chromium carbide and iron, nickel or cobalt, have been patented. This paper gives the results of some of the tests carried out on alloys based on TiC and containing, also, chromium and nickel or cobalt.

Papers have recently been published in U.S.A.[2,3,4] showing that work on similar lines has been carried on in that country. Starting from cemented titanium carbide, the alloys have been developed in a rather different direction. Improvements in oxidation resistance have been

obtained by the addition of tantalum and niobium carbides, and the range of alloys used have been those containing a larger percentage of carbide than those with which we are mainly concerned in this paper.

Methods of Production

The alloys are produced by the powder metallurgy technique using processes which are, in general, very similar to those used in the hard metal industry. These processes are now well known and have recently been described in a number of papers.[5,6,7]

This type of alloy (as is the case with all sintered hard metals) is distinguished from most powder metallurgy products in that a liquid phase is formed during sintering, and exists in equilibrium with the major part of the carbide, which remains solid and forms a framework preserving the original shape in spite of the considerable contraction. The presence of a liquid phase during sintering is responsible for the soundness and high density (approaching the theoretical) of this type of alloy compared with other powder metallurgy products.

Composition and Structure

The range of compositions which has so far been explored has been confined to alloys containing as low as 10% to more than 50% by weight of metal (either nickel or cobalt), the remainder being carbides, mainly TiC with additions of chromium or chromium carbide (Cr_3C_2). It is the relative volume of carbide and metal phases in the alloy which determines its properties. An alloy with, say, 75% TiC and 25% Ni by weight contains 84% TiC and 16% Ni by volume.

The principal carbide phase in the alloys containing chromium consists of a solid solution of Cr_3C_2 in TiC. The solubility of Cr_3C_2 in TiC is over 40% by weight at 1700°–1800° C., but decreases rapidly with fall in temperature. The liquid phase formed during sintering consists of an alloy of nickel or cobalt with carbon, titanium, and that part of the chromium which is not in solid solution in the TiC. It has a freezing range which depends on the carbon and chromium contents and generally lies between 1200° and 1300° C. During solidification and cooling to room temperature most of

1 Engle, E. W., "Cemented Carbides," Symposium on Powder Metallurgy, A.S.M. Publication, 1942, p. 444.
2 Petrowsky, A. R., "The Applicability of Ceramics and Ceramals as Turbine Blade Materials for the Newer Aircraft Power Plants," A.S.M.E. Preprint, Dec., 1948.
3 Hoffman, C. A., Ault, C. M., and Gangler, J. J. "Initial Investigation of Carbide-type Ceramal (80% TiC, 20% Co) For Use as Gas Turbine Blade Material," N.A.C.A. Technical Note No. 1836.
4 Redmond, C. and Smith, E. N., "Cemented Titanium Carbide," A.I.M.M.E. Metals Transactions, vol. 185, Dec., 1949.

5 Brownlea, L. D., Geach, G. A., and Raine, T., "Preparation of Carbides." Symposium on Powder Metallurgy, Iron and Steel Institute Special Report, No. 38, pp. 73–78.
6 Burden, H., "The Manipulation and Sintering of Hard Metals," Ibid., pp. 78–83.
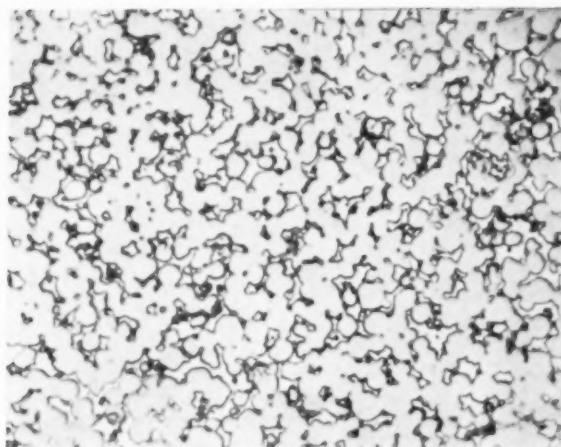7 B.I.O.S. Final Report, No. 1385, "The German Hard Metal Industry."

Fig. 1.—Carbide and metal phases in a fairly high nickel alloy. Electrolytically etched in oxalic acid. × 1,500.

Fig. 2.—Carbide and metal phases in an alloy of medium cobalt content. Etched as above. × 1,500.

the dissolved TiC and some of the chromium and carbon are precipitated from solution.

The structure of the slowly cooled alloys consists, at room temperature, mainly of two phases—small, rounded particles of the carbide phase (a solid solution of Cr_3C_2 in TiC) and a metal phase of large grain-size consisting of nickel or cobalt containing, in solid solution, chromium, carbon, and probably a very small percentage of titanium. One or more other phases may be present depending on the chromium and carbon contents, and on the particular metal used, i.e., nickel or cobalt.

If the chromium content is low and the carbon content is adjusted within narrow limits, two phases only are present, as shown in Figs. 1 and 2. The proportions of the two main phases seen in the micro-structure vary as the metal content is increased. The grain-size of the carbide particles is small, ranging from approximately 2 to 15 microns, but the grain-size of the metal phase is very large, of the order of 1 to 2 mm. Each metal grain contains millions of fine carbide particles arranged in a continuous skeleton.

If the carbon content is high, free carbon is found in the structure in the form of clusters of very fine graphite flakes, precipitated in the metal phase (Fig. 3). The individual flakes are very fine and difficult to distinguish since, in polishing, adjacent carbide grains tend to be dislodged ; because of this dislodgement of carbide grains, however, the cluster formation is easily distinguished. The amount of free carbon is much smaller than appears to be the case in a polished section, and a small amount of free carbon appears to have little effect on the properties of the alloys.

In alloys of the normal carbon content, free chromium carbide (Cr_3C_2) is present when the chromium content exceeds a value which depends on the proportion of carbide present, and the nature of the binder metal (nickel or cobalt). When a small excess of chromium carbide is present it appears as finely dispersed particles in the metal phase (Fig. 4), but when there is a large excess it precipitates as coarse aggregates (Fig. 5).

Titanium carbide forms a eutectic with nickel or cobalt, and there appears to be some solid solubility of TiC in the metal at the eutectic temperature, decreasing rapidly to a very small value at low temperatures. No
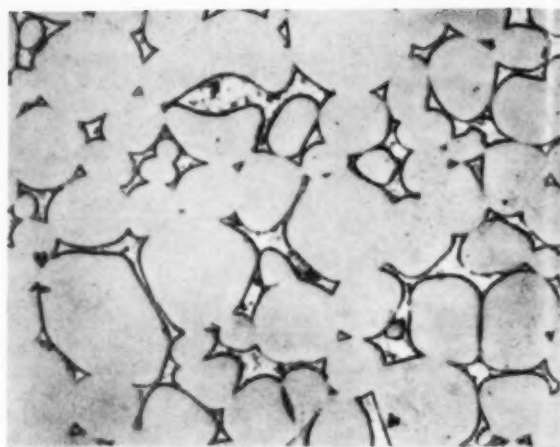
age-hardening has been observed in these alloys and it has not been found possible to change the mechanical and physical properties by heat treatment.

Room Temperature Properties

The specific gravity, hardness, and transverse rupture strength of a number of alloys are given in Table I.

TABLE I.—SPECIFIC GRAVITY, HARDNESS AND TRANSVERSE RUPTURE STRENGTH.

Nominal Composition, Weight %			Specific Gravity	Hardness V.P.N.	Transverse Rupture Strength lbs./sq. in.
Metal	TiC	Cr_3C_2			
20 Ni	74	4	5·80	1400	100,000
30 Ni	63	7	5·90	900	130,000
40 Ni	48	12	6·25	800	180,000
50 Ni	47·5	2·5	6·40	720	230,000
60 Ni	32	8	6·80	560	220,000
20 Co	80	—	5·40	1400	125,000
30 Co	63	7	5·90	1200	115,000
40 Co	48	12	6·29	1180	140,000
50 Co	45	5	6·45	820	230,000
60 Co	32	8	6·88	700	230,000

The specific gravity of pure TiC is 4·91 and that of nickel or cobalt is 8·90. The specific gravity of the alloys increases as the proportion of metal to carbide increases, and substitution of Cr_3C_2 for TiC slightly increases the specific gravity, which varies from 5·15 for an alloy with 10% metal to 6·80 for an alloy with 60% metal.

The hardness of the alloys increases as the proportion of metal phase decreases, as is usual with this type of alloy. Cobalt-bonded alloys are harder than similar nickel-bonded alloys. Variations in chromium content do not affect the hardness appreciably. The hardness of nickel-bonded alloys varies from 560 to 1,400 V.P.N., and that of corresponding cobalt-bonded alloys from 700 to 1,400 V.P.N.

The transverse rupture strength of nickel and cobalt-bonded alloys of comparable composition are similar. In general, if the proportion of metal phase is kept constant, the transverse rupture strength decreases as the Cr_3C_2 content increases. If the ratio of Cr_3C_2 to TiC is kept constant the transverse strength decreases as the proportion of metal phase decreases. The transverse strength is of the order of 130,000 to 240,000 lbs./sq. in.
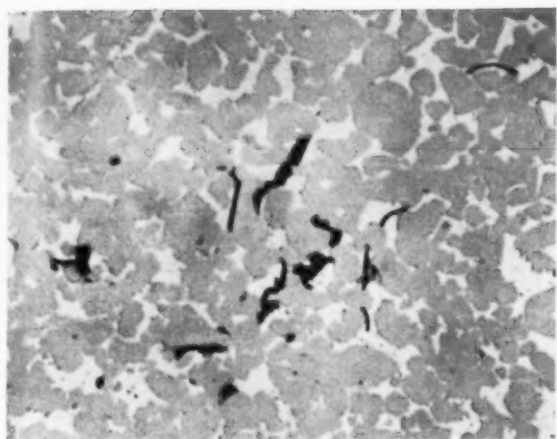
Fig. 3.—Group of graphite flakes in the metal phase of a fairly high nickel alloy. Unetched. × 1,500.

and no measurable plastic deformation occurs before fracture in the transverse rupture test.

Measurements of Young's Modulus have been made, both at room temperature and at higher temperatures, and typical results for this type of alloy are as follows :—

Temperature, °C.	Young's Modulus, lbs./sq. in.
20	$50 \cdot 65 \times 10^6$
700	$38 \cdot 4 \times 10^6$

High Temperature Properties

Oxidation Resistance.—This property was assessed by measuring the gain in weight of test-pieces heated in air under standardised conditions. The specimens were ground on all surfaces, supported on porcelain combustion boats, and heated for 100 hours in air at 900° C. In most cases the scale was strongly adherent but where any scale flaked away this was collected and weighed with the test-piece. The oxidation rate, expressed as the gain of weight after 100 hours at 900° C. varies from 0·0180 down to 0·0040 gms./sq. cm., depending on the composition, and is reduced considerably by the addition of chromium carbide.

The oxidation resistance of these alloys compares favourably with that of many heat-resisting steels. The relatively low rate of oxidation is a result of the forma-tion of a thin, compact oxide layer which adheres strongly to the surface even when it is relatively thick ; this restricts the access of oxygen to the surface of the alloy. The scale is particularly effective on alloys containing chromium.

*Thermal Expansion.*—Measurements of the coefficient of thermal expansion have been made on pieces 2 ins. long in a simple laboratory dilatometer. The results are not of high accuracy, but they show what would be expected from the structures, the coefficient of linear expansion being intermediate between that of TiC $(7 \cdot 1 \times 10^{-6}$ at $20°$–$500°$ C.)[8] and that of nickel or cobalt. The figures obtained vary from $7 \cdot 9$ to $10 \cdot 0 \times 10^{-6}$ per °C. over the range $20°$–$400°$ C. but somewhat higher results $(8 \cdot 9$–$11 \cdot 3 \times 10^{-6}$ per °C.) are found over the range $20°$–$800°$ C. These figures are low compared with most iron and nickel-based alloys and they decrease as the proportion of carbide phase is increased.

Resistance to Thermal Shock.—A few tests have been carried out to compare the resistance to thermal shock with that of other high temperature materials. These tests have been carried out on wedges repeatedly heated locally on the thin edge to temperatures of about 900° C. and rapidly cooled. TiC-based alloys with fairly high percentages of nickel or cobalt have been found to possess resistance to thermal shock as good as, or better than, many other present-day heat-resisting alloys. However, insufficient work has so far been carried out to express the results in quantitative form.

The good resistance to thermal shock is surprising in a material with such low ductility, and must be attributed mainly to the low coefficient of thermal expansion and relatively high thermal conductivity, together with the fact that no structural changes occur in these alloys on heating until the melting temperature is approached.

Creep and High Temperature Fatigue Strength

Creep tests were carried out using the miniature tensile creep-testing machine designed by Harris.[9] Screw-headed test-pieces were used with a gauge length of $1\frac{1}{8}$ ins. and a diameter of $0 \cdot 1785$ in. The creep strain was measured by means of a dial gauge on the stressing beam, so that creep occurring in the test-piece holders

8 Gangler, Robards and McNutt, " Physical Properites of Seven Hot-Pressed Ceramics," *Materials and Methods*, Nov., 1949, p. 77.
9 Harris, G. T., " A Small-Scale Creep-Testing Unit," *Metallurgia*, vol. 34, No. 201, July, 1946, pp. 129–132.
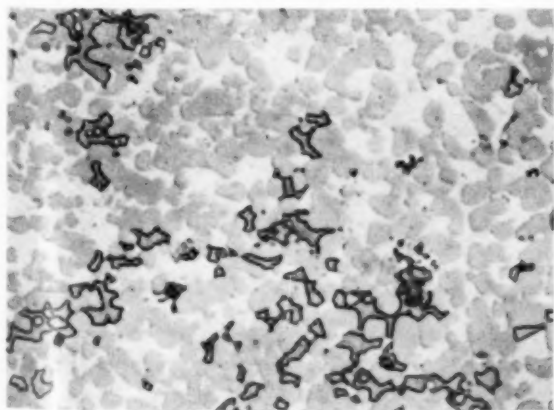

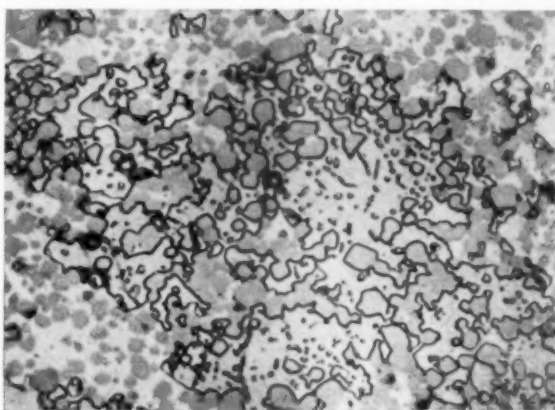Fig. 4.—Free Cr_3C_2 in a high nickel alloy containing Cr_3C_2. Etched electrolytically with 3% KOH. × 1,500.

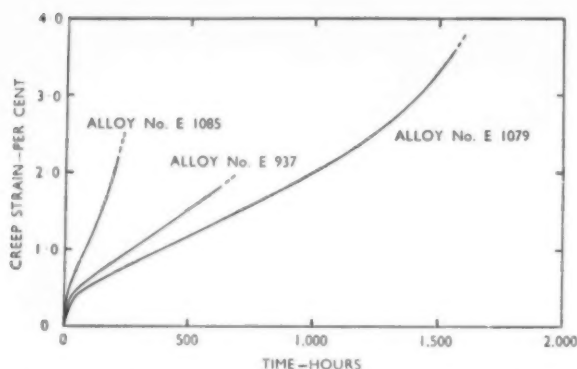Fig. 5.—Coarse crystals of Cr_3C_2 in a high nickel alloy containing more Cr_3C_2. Etched as above. × 1,500.

Fig. 6.—Creep curves at 12 tons/sq. in. at 750° C.

Fig. 7.—Stress v Log Time to Fracture at 750° C.

was also measured. This creep is small compared with that in the test-piece but it should be noted that it is included on the curves reproduced here. A few creep tests carried out on test-pieces of 2 in. gauge length, using more accurate extensometers, confirmed that the miniature test was reasonably accurate.

Almost all creep tests so far have been carried out at 750° C. and typical creep curves obtained are shown in Fig. 6, which gives the curves for three alloys stressed at 12 tons/sq. in. Generally, there is a rather large primary stage followed by a long secondary stage, with a constant creep rate; this is followed by a tertiary stage with increasing rate of creep before fracture. Typical test results, including figures for the secondary creep rate and the total measured elongation, are given for a number of alloys in Table II.

A straight line relationship is obtained when the logarithm of the time to fracture is plotted against the stress for times up to 1,600 hours. Curves for two alloys are given in Fig. 7; the small amount of scatter illustrates the consistency of results obtainable from these alloys. By interpolation the stress for fracture in 300 hours at 750° C. has been obtained for a number of alloys, and values for four of these are given in Table III.

The following conclusions have been drawn from the tests so far carried out.

(1) As the proportion of metal phase is increased, the creep strength is reduced, the secondary creep rate is increased, and the total elongation is also increased.

(2) Cobalt-bonded alloys have higher creep strength, lower secondary creep rate, and higher total elongation than corresponding nickel-bonded alloys.

(3) The addition of chromium to alloys of TiC with nickel or cobalt increases the creep strength considerably, but there is an optimum chromium content above which further increase in chromium results in a decrease in creep strength and total elongation.

(4) A few creep tests carried out at 800° C. and over have given promising results.

(5) There is relatively little scatter in the results and the figures are reproducible.

(6) The creep strength of the alloys tested lies in the same range as that of the best high temperature alloys in use to-day.

A small number of high temperature fatigue tests have been carried out and the results of some of these are included in Table IV. These tests were carried out on rotating beam machines in simple bending with zero mean stress, and the results indicate that high temperature fatigue strength of these alloys is also in the same range as that of other present-day high temperature materials.
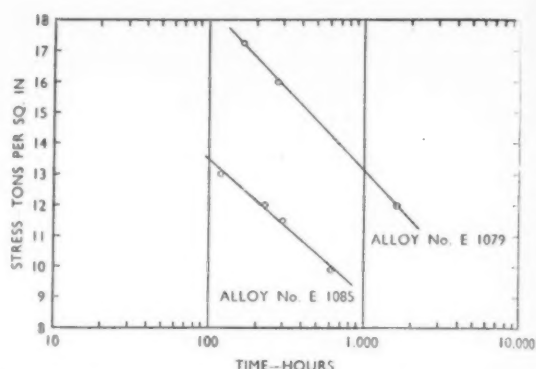
Discussion

The alloys considered here form a distinct group which is related to high alloy steels and the Co-W-Cr-C alloys of the Stellite type. In the sintered carbides, however, the carbide particles occupy a major part of the volume of the alloy and have a dominant role in determining the properties. They have, therefore, high hardness and strength, which is maintained well at high temperatures, low ductility, high Young's Modulus, relatively low coefficient of thermal expansion, and

TABLE II.—CREEP PROPERTIES.

Alloy Number	Tempera-ture ° C.	Stress tons/sq. in.	Secondary Creep Rate in./in./hr. $\times 10^{-6}$	Duration hours	Total Measured Elongation %
E.937	750	12	28·5	671	1·95
	750	14	51	238·1	1·75
	750	15	76	102·6	1·2
	800	12	170	77·7	1·7
E.1085	750	9·9	37·5	608	2·8
	750	11·5	80	295·5	3·2
	750	12	86	230	2·6
	750	13	168	117	2·45
E.1082	750	16	49	308	2·3
	750	17	71	177·5	1·8
	750	18	—	89	1·8
E.1079	750	12	16	1,612	3·7
	750	16	72	276	2·8
	750	17·25	130	166	3·0

TABLE III.—VALUES FOR STRESS FOR FRACTURE IN 300 HRS. AT 750° C.

Alloy Number	Stress for fracture in 300 hrs. at 750° C. tons/sq. in.
E.937	13·4
E.1085	11·4
E.1082	16·0
E.1079	15·9

TABLE IV.—HIGH TEMPERATURE FATIGUE PROPERTIES.

Alloy Number	Temperature ° C.	Stress tons/sq. in.	Cycles to fracture $\times 10^6$
E.744	700	±17	28·6
	700	±19	11·64
	700	±20	0·54
	800	±14	24·0
	800	±15	8·2
	800	±16	6·56
E.1166	750	±13·5	49·05*
	750	±15·0	11·84
	750	±16·0	2·71

* Considered to be premature due to movement in test-piece grips.

relatively high thermal conductivity. The low specific gravity of alloys containing a large proportion of TiC is a useful property, particularly for turbine rotor blades.

The carbides used are very stable and of low solubility in the metal so that the alloys appear to be almost entirely insusceptible to heat treatment. The properties remain unchanged by any heat treatment process so far attempted.

The properties of the alloys can be varied in the way which would be expected from a knowledge of their structure. Increasing the proportion of carbide present :
(1) Lowers the specific gravity.
(2) Increases the hardness.
(3) Decreases the room temperature strength.
(4) Increases the creep strength at high temperatures.
(5) Decreases ductility.
(6) Increases Young's Modulus.
(7) Decreases the coefficient of thermal expansion.

The addition of a small percentage of chromium to Ni or Co-TiC alloys considerably increases their resistance to oxidation and their creep strength.

With a knowledge of the controlling factors, it is possible to select from the range of possible compositions those which are likely to give the best performance in a particular application. For example, if high resistance to oxidation is required, and the creep strength is of less importance, the chromium content can be raised above that required for optimum creep strength. If maximum creep strength is required, and lack of ductility is not objected to, the alloys in the lower range of metal (nickel or cobalt) content can be used.

The range of alloys described here is confined to compositions based on TiC bonded with nickel or cobalt, with the addition of chromium, which is shared between the carbide and the metal. The physical and mechanical properties which can be expected from this type of alloy are fairly clear. Little work has been carried out so far to improve the properties by the addition of further alloying elements, but there is a possibility of further development of these alloys in this direction.

Acknowledgments

New Advisory Panels on Efficient Production Practice

NOMINATIONS have just been received for Advisory Panels representing various sections of the engineering industry. The Road Vehicle and Accessory Manufacturers' Advisory Panel is as follows : H. N. WHITE-HOUSE, Esq. (Austin Motor Co., Ltd.) ; J. SILVER, Esq. (Jaguar Cars, Ltd.) ; J. HARRIS, Esq. (Standard Motor Co., Ltd.) ; L. H. SEWELL, Esq. (A.E.C., Ltd.) ; D. L. CAMPBELL, Esq. (Albion Motors, Ltd.) ; G. MURRAY, Esq. (Pressed Steel Co., Ltd.) ; A. HOSKER, Esq. (Leyland Motors, Ltd.) ; A. T. CHEESLEY, Esq. (Metropolitan-Cammell Carriage & Wagon Co., Ltd.) ; A. E. HUDSON, Esq. (Westinghouse Brake & Signal Co., Ltd.) ; T. BISSELL, Esq. (J. B. Brooks & Co., Ltd.) ; B. STEVENSON, Esq. (Marcroft Wagons, Ltd.) ; C. F. CUNNINGHAM, Esq. (Projectile & Engineering Co., Ltd.) ; A. THOMAS, Esq. (Bromilow & Edwards, Ltd.) ; J. PERCIVAL, Esq. (Fisher & Ludlow, Ltd.).

These industrial experts are to advise on the practical research being carried out by the Production Engineering Research Association of Great Britain, Melton Mowbray, under the direction of Dr. D. F. Galloway, on behalf of the engineering firms represented. Other Advisory Panels now being formed include : Agricultural and Earth Working Equipment Manufacturers' Advisory Panel ; Electrical Equipment Manufacturers' Advisory Panel ; Engine and Turbine Manufacturers' Advisory Panel.

Steel Pipelines

DOLLAR contracts to the value of over £2,000,000 have recently been negotiated between Stewarts and Lloyds, Ltd., and American Oil and Natural Gas Companies, for the supply of many thousand tons of steel pipelines for shipment to the U.S.A. One of the largest orders was arranged through the Anglo-American Oil Co. on behalf of the Standard Oil Co. of New Jersey. The pipes will be manufactured at the Company's works in Scotland, the Midlands and South Wales.

New Yugoslav Metallurgical Institute

A NEW Metallurgical Institute was opened on July 1st, 1950, in Ljubljana, Yugoslavia. It includes, in addition to the usual research departments, a smelting department for the experimental manufacture of different kinds of steel and alloys, etc. There are also workshops for experimental rolling, pressing, casting and drawing of metals, and for testing the reaction of alloys under thermic processes. Special attention is to be paid to the properties of alloys, including their resistance to chemicals.

The British Cast Iron Research Association

October Conference at Ashorne Hill

THE subject of the usual autumn conference to be held at Ashorne Hill in October next has been a matter for discussion. It is anticipated that the report of the General Ironfounders' Productivity Team to the U.S.A., led by Mr. S. H. Russell, will be published by H.M. Stationery Office early in September next, and some aspects of the report will be discussed at the C.F.A. Conference later in that month. By arrangement with the Joint Iron Council, the purely technical aspects of the report will be dealt with at the above Associations Ashorne Hill Conference and will possible occupy Thursday afternoon, October 12th, and the whole of Friday, October 13th. These aspects, which will include metallurgical considerations, foundry sands and moulding practice, melting practice, mechanical equipment and foundry layout, will be dealt with by the appropriate members of the Productivity Team. All the members of the team will be invited to attend on this occasion.

Representatives of member-firms who attend the Conference may spend the nights of Thursday and Friday, October 12th and 13th, at Ashorne Hill. Fuller information will be available from the Association.
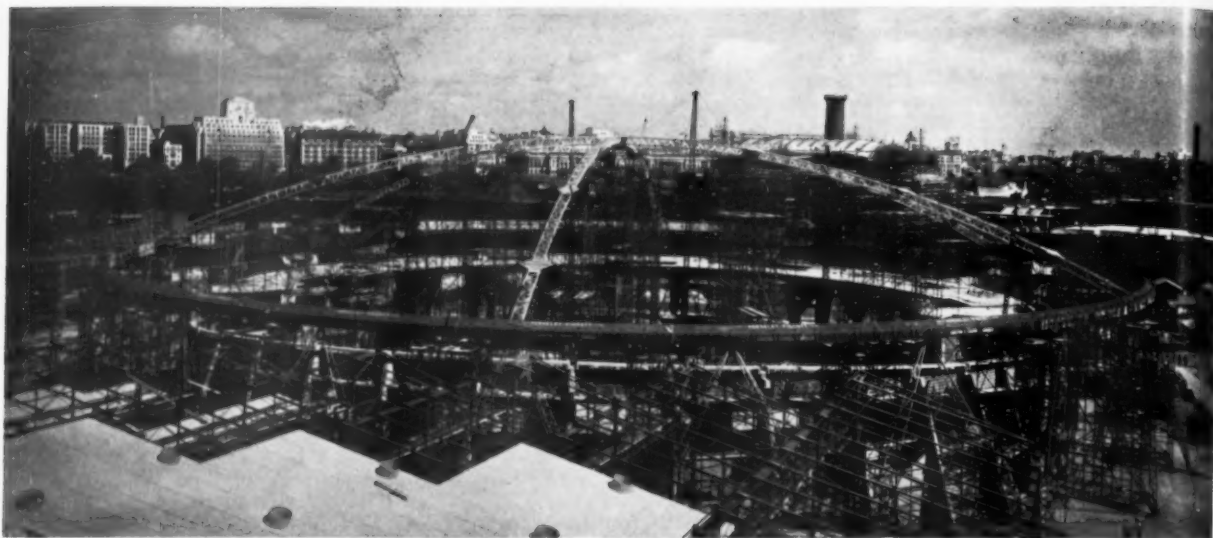
The main structure for the Dome of Discovery in aluminium alloy.

Courtesy of Almin, Ltd.

Outstanding Applications of Aluminium Alloys

Since the war the application of aluminium alloys for structural purposes has made continued and steady progress and has been an important factor in the economy of the aluminium industry. The trend shows no immediate signs of being checked and in this article three examples are described in which the inherent properties of the aluminium alloys have been responsible for their choice.

The Dome of Discovery

A STRIKING feature of the Festival of Britain will be the giant Dome of Discovery which will " house the story of British predominance in discovery and exploration, not only by land and by sea, but also into the very nature of the living world and the universe." In addition to being the largest dome in the world—the whole structure has an overall diameter of 365 ft. and a height, from ground level to the centre of the Dome, of 90 ft.—it is, by far, the largest single structure ever to be built of aluminium. The method of construction is unique, the main arch ribs being arranged as two great circles in three directions to form a triangulated structure. Resting on a steel ring-girder, the whole of the Dome structure, comprising arch ribs, rafters, purlins, roof sheeting and canopy is of aluminium. Also of aluminium is the apron which encloses the underside of the dome and projects up to 75 ft. beyond the perimeter of the main exhibition hall, on one side, to form a huge canopy over the main entrance. In all some 232 tons of aluminium will be used.

S.M.D. (Structural and Mechanical Development Engineers, Ltd.), the development and construction company of the Almin Group, is responsible, as sub-contractors, for the manufacture of the main girders, and for the complete apron structure and the canopy.

Six of the main ribs radiate from the centre of the dome, while six others form two interlocking triangles circumscribed by the periphery of the dome. Each of these ribs is built up of eight sections of various lengths, the largest being 57 ft. long and weighing 12 cwt. The intermediate ribs do not follow great circle lines exactly but are shaped to form smooth curves which run generally with the main ribs. Between the main ribs and the intermediate ribs run the rafters, of a special aluminium alloy " I " beam extrusion. Each triangular opening between ribs and rafters is spanned by extruded aluminium alloy " top-hat " purlin sections. The covering sheet will be supplied flat and pulled down to the correct curvature on the purlins.

The main ribs are triangular lattice girders built up from seven extruded sections, the problem of the accurate bending of all the sections to the required radius being solved most successfully by the two material suppliers, I.C.I., Ltd., and Southern Forge, Ltd. Extra large drilling and assembly jigs enabled the required building accuracy to be achieved whilst the problem of cold driving $\frac{5}{8}$ in. diameter rivets in A.W.5 alloy ($3 \cdot 5\%$ Mg, $0 \cdot 5\%$ Mn) was solved in co-operation with Broom & Wade, Ltd., by designing and making special single-blow pneumatic squeeze rivets capable of exerting 25 tons pressure. Special portable band saws were developed for accurately trimming the ends of the main rib sections after jig assembly.

All the structural sections are of extruded heat treated A.W. 10B alloy (1% Si, $0 \cdot 6\%$ Mg, up to 1% Mn) with a $0 \cdot 1\%$ proof stress of 15 tons/sq. in., a U.T.S. of 18 tons/sq. in., and an elongation of 10%.

Below the dome, and between its perimeter and the wall of the exhibition, is the apron, in the form of a huge

inverted truncated cone forming a great sloping canopy all round the building. One of the problems presented by this part of the project, the forming of purlins to different radii between 101 ft. and 166 ft. in 3 ft. 3½ in. increments, at an angle to the natural bending plane of the section, has been overcome by the development of a new process by Southern Forge, Ltd.

Extending all round the dome, beyond the main structure, is a canopy about 8 ft. deep consisting of pressed aluminium alloy troughs, supplied by the Warwick Production Co., bolted to the outer edge of the ring girder and sheeted on top with 18 S.W.G. A.W. 4C sheet. The flashing around the outer edge of the canopy is of 20 S.W.G. aluminium.

Telescopic Gangways for New Ocean Terminal

The new Ocean Terminal at Southampton was opened by the Prime Minister on Monday, July 31st. Designed specifically for the reception of the Cunard White Star "Queen" vessels, the building is over 1,200 ft. long and provides boat-train platforms, Custom halls, waiting-rooms, buffets, writing rooms, telephone rooms, information kiosks, lifts, escalators and sight-seers gallery.

The normal type of gangway, which would need to be lifted into position by crane, was not considered a suitable proposition, particularly in high winds. It was also felt that they would not match the "luxury" character of both ship and terminal building. The solution was found in totally enclosed gangways which can be slewed, buffed, extended and telescoped. To serve all classes of passengers, three gangways, each consisting of a shore station and two telescopic walkways, ingoing and outgoing, have been provided.

To accommodate various ships the gangways are moved along the quay and the walkways can rise and fall to suit the state of the tide, and rotate sideways to accommodate fore and aft movement of the berthed ships. When not required they can be swung back parallel to the quay to be clear of the cargo crane.

Aluminium alloy was chosen for the structural material in order to minimise the load on the shore structure and to reduce the size of the

operating mechanism, which otherwise threatened to make the whole unwieldy. The project was carried out by Structural and Mechanical Development Engineers, Ltd.

The outer and inner gangways each weigh 30 cwt., the former being 40 ft. × 4 ft. 6 in. × 8 ft. and the latter, 43 ft. × 3 ft. 9 in. × 7 ft. 6 in. with a total fully extended length of 68 ft. Special extrusions are incorporated in the design to ensure stiffness and lightness in weight and the floor is made by sandwiching corrugated sheeting between two flat sheets top and bottom, connection being made to the top sheets with the crest of the corrugations and to the bottom sheet with the trough. A rubber coating cemented on, and carrying wooden battens at intervals completes the flooring. The telescoping motion is electro-mechanical through gear boxes and roller chains.

The turrets transmitting the electro-hydraulic slewing and buffing motion to the gangways are necessarily of extremely robust construction. The sides are made from plate box girders of ¼ in. material, supermounted by a box beam which carries the journals for the two hydraulic slewing rams together with the shaft by which

Courtesy of Almin, Ltd.

Telescopic gangways for the new Ocean Terminal at Southampton. Inner and outer gangways, showing construction, before sheeting.

Courtesy of British Aluminium Co., Ltd.

" Dyson-Express " 100-Seater semi-trailer coach coupled to Thornycroft motive unit.

the turret is suspended and which, together with another shaft at the base of the turret frame, form the journals for the rotary movement. The huge pin on which the gangways are buffed is situated at the base of the turret, and a shaft at the upper part of the turret carries the cylinder-end of the hydraulic buffing rams disposed either side of the gangway. A sheet metal joining on the turret covers the buffing rams.

Each turret weighs 1¼ tons, the shore-housing 9 tons, and the telescoping gear 5 cwt., the total completed weight being 17½ tons. The aluminium alloys used are : A.W. 10B for the structural sections; A.W. 5 for sheeting; and A.W. 6D for rivets of which, it is noteworthy, each structure contains 100,000 besides 12,500 bolts.

An ingenious system of interlocking controls has been devised to ensure the maximum safety of the equipment and operators. Until the anchors provided to lock the mechanism to the quay rail are secured, it is only possible to travel the structure along the quay side—no hydraulic motor is possible. Once the anchors are secured, it is impossible to travel along the quay and power is available to manoeuvre the gangways one at a time. Other interlocks prevent movements being made in the wrong order and provide safety alarms in case the maximum extension is approached with the gangways coupled to the ship.

Dyson-Express
100-Seater Semi-Trailer Coach

Oil companies for some time past have favoured semi-trailer coaches as an efficient and economical means of personal transport in the oilfields. Not only is it possible to transport a large number of passengers, but

the tractor units after hauling the coaches to their destination can be uncoupled and used with other semi-trailers for general transport.

The Kuwait Oil Co., Ltd., needing to augment and replace its existing fleet of passenger transport vehicles, placed a contract for a number of 100- and 80-seater semi-trailer coaches with R. A. Dyson & Co., Ltd. This concern arranged to sub-contract the building of the bodies with the Express Motor and Body Works, Ltd., and the vehicles now produced are the result of the close co-operation between these two companies, assisted by the engineering staff of the Kuwait Oil Co., Ltd., and in consultation with the British Aluminium Co., Ltd. Although the order was not placed until the end of November, 1949, the first two coaches were ready for shipment in early June—a very creditable effort in view of the numerous design and constructional problems to be overcome.

Judged by British standards, the size of the 100-seater coach is colossal. The semi-trailer itself is nearly 40 ft. long and, coupled to the Thornycroft VN tractor, the overall length is 52 ft. As the vehicles will operate in a very hot climate, insulation is necessary and only comparatively small windows are provided. Further the coaches may have to traverse rough ground so that a high ground clearance is essential.

The chassis frame is of the double cranked pattern, being upswept at the front and over the rear axles. The main difficulty has been to design and construct a chassis frame which, although comparatively shallow, will have a very small deflection under load. Any
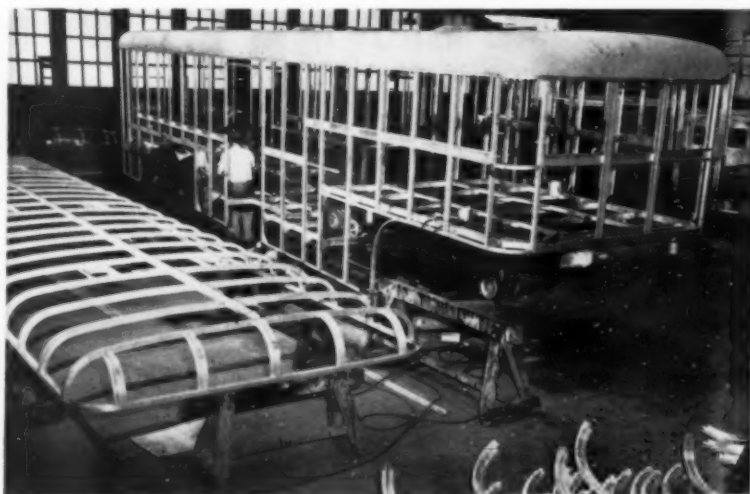
Courtesy of British Aluminium Co., Ltd.

Main assembly showing all the framework, also the roof framework assembled prior to being placed in the jig for riveting, skinning, insulation and electrical installation.

excessive flexing of the chassis frame would set up strains in the bodywork which, in time, might have a harmful effect on the structure. For this reason, most of the main frame longitudinals are doubled, and instead of single members there are two heavy channels back to back with a central reinforcing plate in between them at the points of maximum stress. The prototype chassis frame was subjected to very stringent tests to ensure that theory and practice coincided and it was found that the actual deflection was well within the required limits.

In view of the size and strength required in the construction it was particularly satisfying to achieve the relatively low body weight of 2¼ tons. As would be expected, in view of the low weight/size ratio, the coach is largely built of aluminium alloys. In selecting the materials used, particular attention was paid to the stress likely to be experienced with a vehicle of such large dimensions and, although the body weight is comparatively low, no sacrifice has been made as regards strength. All structural sections are in precipitation hardened B.A. 25 alloy (0·7% Mg, 1% Si) which has a minimum U.T.S. of 18 tons/sq. in. The flooring is also in this alloy, in the form of British Aluminium's P-G-P (positive grip pattern) treadplate. For panelling, half-hard commercial purity (99%) aluminium sheet is used, whilst gusset plates are in B.A. 21 (2·25% Mg, 0·4% Mn). The cant rails (A.D.A. C/3 section) in the side of the body are of special interest in that they have been extruded in one length of 36 ft. 3 in. and specially drilled so that, whilst being secure, they permit interchangeability of the roof. All main window frames are also interchangeable and can be replaced very easily.

The body bearers are of aluminium alloy channel section and are fastened to the main chassis by high tensile bolts and locking nuts, "Ferodo" packing being used between bearer and chassis. D.T.D. 303 rivets are used for making joints between structural members and for skinning "Durolac" being applied between all mating surfaces.

Developments in Refractory Materials

Significant improvements in refractories in recent years have contributed considerably to the high output of the iron and steel industry. The British Ceramic Research Association and The British Iron and Steel Research Association have assisted manufacturers in these improvements which have resulted in furnaces being kept in operation for longer periods.

IT has been said that there is no such thing as a good firebrick ; its qualities can only be assessed in relation to the function which it is to perform. This is especially true of the steel industry where the phenomenal technical developments of the past two or three decades have led to an increasingly stringent selection of refractory materials. In particular, the value of refractories having an alumina content higher than that of the raw materials indigenous to these islands, has come to be recognised. Several firms who have established well-known grades of refractories have been justified in increasing their plants to manufacture high alumina bricks to serve the steel and certain other industries. Of these, mention may be made of the large works at Neston, Cheshire, which have been built for the Morgan Crucible Co., Ltd., expressly for this purpose. This plant will also be producing shortly a range of high temperature insulating bricks capable of withstanding temperatures up to 1,600° C.

Another typical example of developments by manufacturers to meet the needs of the iron and steel industry is that by Messrs. Gibbons (Dudley), Ltd., who, since the summer of 1949, have had in full production, at their refractory firebrick works, a new tunnel kiln, designed and built by their associate company, Messrs. Gibbons Bros., Ltd. The layout of this plant consists of a three-bay building housing a tunnel dryer 160 ft. long, a tunnel kiln 310 ft. long, two loading tracks, two unloading tracks, and a rubber belt conveyor for the transfer of the fired goods from the unloading tracks to various sections of the stocking bay. The unfired shapes are brought from the adjacent moulding floors to the loading bay by a 2-ton capacity electric truck, the goods being just sufficiently dry to enable them to withstand setting on the kiln trucks without squatting. A very

A view of the large works at Neston, Cheshire, recently built for the Morgan Crucible Co., Ltd.

large range of sizes is handled from small specials, regenerator tubes, muffle chamber fronts, to large and solid specials 8 in. thick, weighing up to 155 lb. Weekly production in this plant averages about 207 tons burnt weight. The main zone in the kiln is constructed in 95% silica brick and designed for a temperature up to 1,500° C. The goods are fired to Seger cone 12 on a 157 hour cycle. Cooling of the goods takes place in 38 hours.

All the main manufacturers of refractory materials for the iron and steel industry have found it necessary to make plant extensions and to carry out investigations into the behaviour of these materials. General Refractories, Ltd., for instance, have recently built a new central research laboratory complete with pilot plant, the purpose of this extension being to permit of an increased investigation into the behaviour of refractory materials and to evolve means of producing linings capable of withstanding ever-increasing demands.

Loaded truck being transferred to tunnel dryer by means of electric transfer truck.

Fired trucks being unloaded on to a rubber belt conveyor at the works of Messrs. Gibbons (Dudley), Ltd., where this new plant is installed.

High Temperature Insulating Bricks

In recent years fuel economy has become a permanent feature of industry but, in addition, there is an increasing awareness of the potential savings of time which are rendered possible by the use of refractory materials of low conductivity and low thermal capacity. Furnaces incorporating such materials exhibit extremely low thermal inertia and the minimum proportion of heat is absorbed by the fabric.

The thermal insulation of furnaces operating at very high temperatures has been scarcely practical because refractories for lining such furnaces, capable of resisting the increased mean temperature due to the reduction in the heat dissipated through them, were scarce.

Much attention has been given recently to the manufacture of suitable insulating bricks and in addition to the range for high temperature work to be produced by The Morgan Crucible Co., Ltd., at their new works at Neston, and by other firms, investigations carried out by Thomas Marshall & Co. (Loxley), Ltd., have shown that vermiculite can be successfully used for this purpose. By the end of 1947 that company had solved the main problems of the manufacture of insulating brick, using vermiculite as the main constituent. Since that time the company has been producing bricks and tiles, having designed its own type of furnace for the exfoliation process and installed specialised forming and kilning plant. Recently output was at the rate of 10,000 $9 \times 4\frac{1}{2} \times 3$ in. bricks per week with facilities for stepping up output to meet market requirements.

Vermiculite bricks, which are remarkably consistent in their properties, as produced by Kingscliffe Insulating Products, Ltd., a subsidiary of Thomas Marshall & Co., have a maximum safe working temperature of 1,100° C. and can be made in bulk densities from 24–28 lb./cu. ft. according to the application for which the bricks are intended. The properties of these bricks are at least equal, and often superior, to a porous firebrick of the same density, but in the lower density range their very

low heat capacity is of great importance. An advantage of the vermiculite brick is the fact that it can be produced in quite large slabs—e.g., $18 \times 9 \times 4\frac{1}{2}$ in., or almost any rectangular shape, without resort to cutting or machining after forming. On the other hand, if the bricking should be particularly complicated and the bricks or slabs have to be shaped, they can be cut or drilled easily with ordinary woodworking tools.

High Thermal Conductivity Refractories

While the thermal insulation of furnaces is of great importance in the conservation of heat and, therefore, in their economical operation, high thermal conductivity materials are also invaluable in reducing operating costs in indirect and semi-direct fired furnaces. Through their use as the heat transfer medium, it is possible to maintain working chamber temperatures although reducing combustion chamber temperatures, with resulting fuel economy or, conversely, to obtain a greater heat flow to the working chamber with the same combustion chamber temperatures—thus increasing production. Silicon carbide refractories, for instance, as represented by Carbofrax, has a thermal conductivity about ten times that of fireclay, and since it does not soften or fuse below its dissociation temperature, which is about 2,200° C., and maintains a relatively large percentage of its cold strength in brick form, it is used for combustion linings, combustion arches, and for many specialised purposes where their special properties make them economical. In a variety of shapes this material is used in the assembly of hearths, muffle walls, batch and continuous furnaces, heat treatment furnaces, etc.

In general, however, the properties sought in refractories for the iron and steel industry are a combination of characteristics which vary with the particular application, but the most important are high refractoriness, resistance to attack of molten slag or iron, high mechanical strength at elevated temperatures, high thermal conductivity, and low after contraction, and brief

reference to developments in particular applications will be of interest.

Blast Furnaces

There has been continuous improvement in the quality of firebrick for blast furnaces : much attention, for instance, has been given to the importance of gas attack resistance, and some furnaces have operated as long as 10 years before blowing out for relining, a typical example being the No. 1 blast furnace at Clyde Iron Works, which was blown out early in 1949, after producing more than $1\frac{1}{2}$ million tons of pig iron since it was blown in during the spring in 1939. The refractories used for the hearth, bosh and inner wall contained 42% alumina as the main constituent and the stack refractory 33–35% alumina ; they were supplied by J. G. Stein & Co. Ltd.

In order to meet the requirements of modern blast furnace operation and realising the necessity of producing a lining superior to firebrick, plants have been put in operation for the manufacture of carbon blocks, the characteristics of which have proved to be the most suitable material so far devised. The early use of carbon linings was restricted to the hearth and bosh, but last year two blast furnaces at Appleby-Frodingham Steel Co., all-carbon lined, were put into operation and a third this year. This refractory carbon is made by Carblox, Ltd., a joint subsidiary of Thomas Marshall & Co. (Loxley), Ltd., and The Morgan Crucible Co., Ltd., and results with these furnaces are reported as being excellent. They are working easily and without trouble. The first furnace is yielding 15% more output of iron, the second 10% more as compared with the operation of the same furnaces when they were previously lined with firebrick. This is partly due to the absence of any hold up due to erratic and uneven working, but it is mostly due to the increase in furnace volume by installing a thinner lining of carbon for the hearth wall and bosh than was used with firebrick. The additional cost of the Carblox in the first all-carbon furnace is stated to have been covered by the first 13 months operation of the furnace.

Recent examinations of furnaces only partially lined with carbon show that the carbon bricks forming the hearth walls and the boshes are mostly as good after full campaigns as when installed.

The stacks of the above three all-carbon lined furnaces are insulated and it is expected that this procedure will be adopted as standard once the success of carbon as the ideal blast furnace refractory lining has been confirmed in practice. This confirmation is well on the way as pre-fired Carblox alone has been supplied for 27 blast furnaces in this country and abroad.

Henry Foster & Co., Ltd., an associate of General Refractories, Ltd., have in operation a plant for the production of pre-fabricated carbon block, carbon ramming mix and carbon jointing paste and many furnaces are now being successfully operated with Foster carbon linings, since the characteristics of carbon not only include high refractoriness, resistance to attack of molten slag and iron, high mechanical strength at elevated temperatures, and high thermal conductivity, but also the valuable characteristic of not being wet by molten slag or iron.

Open Hearth Furnaces

Developments in the design of open-hearth furnaces and their operation, with a view to quicker melting rates,
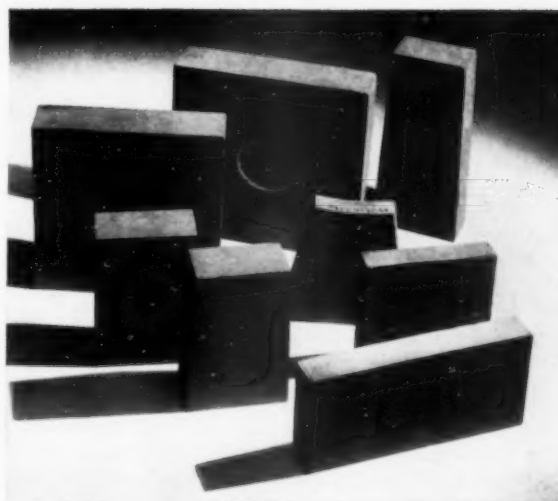
A selection of " Carblox " standard shapes.

has been governed to some extent by the properties of the roof material available. Silica is predominantly used and efforts have been made to increase the life of roof blocks by more careful grading and maintaining control of both grain size and composition. Mechanical pressing of roof blocks, by reducing warpage, has increased structural stability, which is conducive to longer operational life.

Roofs capable of withstanding higher operational temperatures have been met with basic material in the all-basic furnace. Fired chrome-magnesite roof blocks are in regular use, and chemical bonded chrome-magnesite and chemically bonded magnesite-chrome bricks for other positions in these furnaces. The raw material position has an important bearing on the successful application of basic materials in the furnace ends, but much work has been carried out by several manufacturers, and particularly by the research associations B.C.R.A. and B.I.S.R.A., and considerable progress made in developing the necessary characteristics by bricks for this type of furnace.

The development of metal clad bricks is proceeding and General Refractories, Ltd., have acquired the sole rights in this country, to produce a fully metal cased basic brick. Known as the " Ferroclad " this is a chemically bonded brick on which a metal case covering all sides is securely anchored during the processing of the brick. The operation is carried out on a hydraulic press working at high pressures. There are three grades of this brick—viz., chrome, chrome-magnesite and magnesite. Results of trials during recent months are very promising, particularly in connection with hot patching.

Electric Arc Furnaces

An important development is the dolomite brick for basic arc furnaces. It is an entirely home produced brick and is being used with considerable success for the walls in many of the larger basic electric arc furnaces. Chrome-magnesite bricks are being successfully used and, as with open hearth furnaces, the object of all basic furnaces is to increase the rate of working. Much progress, however, has been made in the design and

manufacture of roof blocks for electric furnaces. In addition to silica bricks, there has been development in the use of Sillimanite for this purpose and some service reports are of a high order. A refinement has been the introduction of correctly shaped blocks for the electrode hole assembly.

While significant improvements in refractories during the past few years have greatly assisted the iron and steel industry, there can be no relaxation as furnaces must be kept in operation for longer periods, this is only possible by continued developments of suitable refractories.

Hearth Breakout at Scunthorpe *

THE third all-carbon furnace at the Appleby-Frodingham Branch of The United Steel Companies, Ltd., Scunthorpe, experienced a hearth breakout on July 24th. In view of all the attention which has been directed to the experimental use of carbon refractories at Appleby-Frodingham it is felt that this breakout should be reported.

Owing to limitations of site and peculiarities of foundation construction, very drastic modifications to regular Appleby-Frodingham hearth construction had to be adopted. Full examination of the breakout by the Research and Development Department of The United Steel Companies, Ltd., indicates that there was insufficient hearth carbon to chill the metal known always to penetrate joints. Because of the short hearth jacket and, therefore, the short chimney wall, this iron, still liquid at the bottom of the hearth, found its way through the concrete foundation block and under the hearth jacket. The furnace was off for 38 hours.

This breakout in no way alters any of the views or theories held by The United Steel Companies in relation to hearth carbon, if anything it enhances the value of the experiments in hand. To enable proper perspective to be taken of this breakout it should be pointed out that 2,910,920 tons of pig iron have been manufactured on carbon hearths with no trouble whatever. The corresponding tonnage of iron made before the introduction of carbon was accompanied by 14 breakouts.

* Communication from The United Steel Companies, Ltd.

Staff Changes and Appointments

MR. W. J. S. ROBERTS, B.Sc., F.I.M., has been appointed Chief Metallurgist at the new cold reduction plant and tinplate works of The Steel Company of Wales at Trostre. A " Folland " scholar in metallurgy at the University of Wales, Mr. Roberts obtained his B.Sc. with 1st Class honours in metallurgy in 1927 and after a year's post-graduate course was engaged by the Straits Trading Co., Ltd., at their smelters in Singapore and as works assistant in tin smelting at Penang. In 1934 he joined the Midlands branch of Messrs. Baldwins, Ltd., and in 1937 the Richard Thomas & Co., Ltd., at Llanelly from where he moved to the Ebbw Vale works of Richard Thomas and Baldwins where he has remained until taking up his present appointment.

MR. R. A. MILLER has been appointed General Manager (technical) and MR. R. G. DUCKETT Technical Sales Manager of Foundry Services, Ltd.

MR. J. A. PARDOE has left D. Napier & Sons, Ltd., Liverpool, to take up an appointment as Metallurgist in the Department of Atomic Energy, Springfields Factory, Salwick, Nr. Preston.

MR. M. F. DOWDING, M.A., A.M.I.Mech.E., has bee appointed Engineering Sales Manager of Davy and United Engineering Co., Ltd. Mr Dowding received his engineering training at Cambridge University and has been with Davy and United since 1946 when he returned from service in the Army. Until recently he was the Rolling Mill Research Engineer and has just returned from an extended tour of the U.S. steel industry. He is a member of the Rolling Committee of the British Iron and Steel Research Association.

MR. GORDON H. FIELD, C.B.E., has resigned his position of Director of Research, Aluminium Laboratories, Ltd., Banbury, as from August 31st, 1950. Mr. Field has been on the technical side of the aluminium industry for close on 35 years, 20 of which have been spent with the Aluminium, Ltd., group of companies. It is not Mr. Field's intention to retire from active participation in the work of the industry, but to make his experience available in a consulting or similar capacity. During his connection with the aluminium industry he has served on a large number of Government and British Standards Institution committees, and was honoured with the award of the C.B.E. in the King's Birthday Honours last year.

MR. G. F. ANDERSON has resigned the office of Secretary to Climax Molybdenum Co. of Europe, Ltd., because of the increase of his activities in other directions and MR. H. L. RICHARDSON has been appointed to succeed him.

MR. F. T. PULLIN and MR. W. L. CAVE have been appointed Manager and Assistant Manager, respectively, of R.T.S.C. Exports, Ltd.—the company jointly formed by Richard Thomas & Baldwins, Ltd. and The Steel Company of Wales, Ltd., to deal with the exports of mild-steel flat-rolled products (coated and uncoated) manufactured by them and their subsidiaries.

MR. N. HASLAM has been appointed Metallurgist at the British Iron & Steel Research Association, Sheffield.

MR. W. K. B. MARSHALL has resigned his post with the Welding Research Association to take up an appointment with Messrs. Rockweld, Ltd., Croydon.

SIR W. T. GRIFFITHS, Vice-President of the International Nickel Company of Canada, Ltd., has been re-elected a Director of the Company.

Nyanza Mines

THE Colonial Development Corporation is forming a new Company, to be known as Nyanza Mines, Ltd., to develop and bring to production the Macalder Mine which lies near the shores of Lake Victoria in South Western Kenya. The mine is a copper-gold proposition with the possibility of subsidiary zinc being recovered at a later date. Mr. D. J. Rogers, B.Sc., A.R.S.M., M.I.M.M., has been appointed General Manager and is now on his way to East Africa.

Initially an extensive programme of diamond drilling combined with a geological and geophysical survey will be carried out to determine the mineral resources of the property. At the same time metallurgical test work will be conducted in a pilot mill, on which erection will commence immediately, with the object of deciding the most economical method of ore treatment and the design of the final dressing plant.

The Mechanism of Sintering

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The present knowledge of the mechanism of sintering is reviewed. Evaporation-condensation, surface diffusion, volume diffusion, viscous flow and plastic flow are considered. Sintering in the presence of connected pores is taken as a first stage and sintering in the presence of isolated pores as a second stage, the two stages being dealt with separately.

COMMON usage gives a wide range of meaning to the term " sintering." We shall define sintering as the compound process of the adhesion at elevated temperature of solid particles in contact with one another and the change in dimensions of the compact so-formed. Our attention will be confined to sintering in the absence of a liquid phase.

The sintering process is of great technological importance ; many materials—e.g., metals, glasses, ceramics and coals are fabricated by it. Apart from purely scientific interest, an understanding of the mechanism of sintering is highly desirable because all sintering techniques used commercially ought to be based on sound theory so that optimum results be obtainable. In recent years, a considerable number of papers devoted to the mechanism of sintering have been published. The purpose of this paper is to collect together and examine the important contributions in order to indicate the present state of knowledge.

It is now widely accepted that sintering occurs as a result of the excess free energy of the powder over that of the dense solid, by virtue of the different surface areas of the two states. However, as will be seen below, the *mechanism* of sintering remains a subject of considerable controversy.

Large-scale and very rapid movement of material has to be explained.* Possible agencies for this movement are evaporation-condensation, surface diffusion, volume diffusion, viscous flow and plastic flow (" viscous flow " denoting Newtonian flow—i.e., flow like a liquid, and " plastic flow " referring to flow when a yield stress is involved). Recrystallisation frequently occurs hand-in-hand with sintering, but it is a separate phenomenon.

Sintering in the presence of connected pores will be called the first stage and sintering in the presence of isolated pores will be called the second stage. In the first stage, evaporation-condensation and surface diffusion could cause shrinkage of a compact if there be a net movement of material from the surface regions to the interior, and volume diffusion could cause shrinkage if lattice vacancies move from the interior out through the exterior surface of the compact. Shrinkage could also result from viscous and plastic flow. In the second stage, on the other hand, evaporation-condensation and surface diffusion could only spherise pores and could *not* cause shrinkage. Again, volume diffusion could bring about shrinkage if lattice vacancies pass the exterior surface. Shrinkage would result from viscous and plastic flow. Shaler[1] and others have emphasised the division of the

subject into two stages and it is artificial only insofar as the stages no doubt overlap in time to a considerable extent, but this does not invalidate their separate treatment. If it be proved that the same mechanism is predominant in both stages, the division will lose its usefulness, but until such a time arrives (if ever) it is convenient to retain it.

The first part of this paper is concerned with the first stage ; different concepts of the " driving force " are referred to and then each possible mechanism of the sintering is dealt with. The second part is concerned with the second stage ; here we shall be concerned mainly with sintering *in vacuo*, since the presence of gas in closed pores is an unnecessary complication for the present purpose. Both parts finish with a short note written in retrospect and drawing attention to the important matters involved.

A concise and systematic account of the phenomena occurring during sintering has been given by Rhines[2] (1946). This review deals mainly with work published since Rhines' paper and prior to the beginning of 1950.

PART I—FIRST STAGE OF SINTERING—CONNECTED PORES

I—General

The primary concern of this paper is the mechanism of sintering ; in other words, we are concerned with the processes by which material moves during sintering. Nevertheless, we must first refer to the reasons for the operation of these processes.

It is a matter of experimental observation that there is a reduction of total surface area when particles sinter together. The corresponding reduction in total surface energy takes the system to a lower energy state. This lowering of the total free energy is the factor without which sintering would not proceed, and which is commonly and loosely referred to as the " driving force."

Let us consider for a moment the sintering together of two equal spherical particles, for example. It will be seen that the configuration of the surface of the system is basically important for sintering to proceed, no matter which possible mechanism may occur. It is the surface configuration which would give rise to differential evaporation in the evaporation-condensation mechanism and to concentration gradients for surface and volume diffusion. Again, in the case of viscous and plastic flow, the material would be moved under the influence of surface tension, the magnitude of which, at different points in the surface, depends on the surface configuration. In the greater part of the work referred to in this paper, the surface configuration is involved in the ways just mentioned.

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* e.g.—The density of a block of slip-cast aluminium oxide (melting point 2,050° C.) increased from 2·45 to 3·74 gm. cm.³ after 2 hours at 1,735° C. (oxidising atmosphere) ; in this case, the corresponding porosity change was from 39 to 7% (R.A.E. data).

Metals and non-metals are not known to exhibit strikingly different rates of sintering, conditions being otherwise similar.

Attempts at a rather different approach to the driving force early in the first stage of sintering have recently been made. Attention has been given to the force acting between slightly separated surfaces of the same material. Bangham[3] (1947) considered qualitatively the inter-atomic forces near the contact of two spheres, and predicted that there would be a resultant attractive force between them. Shaler[1] (1949) reported that he had calculated the force of attraction between slightly separated copper surfaces. He had used the Sommerfeld model[4, 5], in which a metal is considered to consist of a lattice of positive ions surrounded by an electron cloud whose density is constant except near the surfaces, where it falls off rapidly. If a second metallic body be close enough to the surface of the first, the electron cloud density increases in the opposite surfaces and the clouds of the two bodies join, producing a force of attraction between the bodies, since each body is attracted to the electron cloud which extends between them. Shaler's values (e.g., that the attraction between copper surfaces separated by 120 Angstrom units is 30,000 lb. in.$^{-2}$) are too large to be plausible[1a], but calculations of this type are of extreme interest from the point of view of sintering of metals.

If these long-range attractive forces between particles be real, large stresses would occur at the areas of contact. It is clear that viscous or plastic flow might readily be brought about. The possible large stresses have not as yet been taken into account in treatment of the other mechanisms. However, it must be emphasised that this concept of a strong attractive force is not on firm ground at present and further determination of the forces between particles is desirable before sintering theory accepts it.

It is not proposed to go into the inter-relation of surface tension and surface energy in solids, nor their nature. Readers are referred to the recent contributions to the subject listed as References 6, 7 and 8.

We now pass on to detailed consideration of mechanisms. The possible ones for the first stage of sintering were enumerated in the introduction; they are evaporation-condensation, surface diffusion, volume diffusion, viscous flow and plastic flow. (It is convenient to stress here that the next two Sections, on evaporation-condensation and surface diffusion respectively, are appropriate to both the first stage and also *sphering of pores in the second stage*.)

II—Evaporation-condensation in the first stage

An idea of the rate of transfer of material from one part of a compact to another by an evaporation-condensation process is obtainable as follows. The rate at which a material is transferred from one surface above which the equilibrium vapour pressure is p_1 to a second surface above which the equilibrium vapour pressure is p_2 is calculable from kinetic theory. The ratio of the equilibrium vapour pressures p_1, p_2 above spherical surfaces of the same material of radii of curvature r_1, r_2 respectively was deduced by Kelvin. If the first surface be flat (Fig. 1), $r_1 = \infty$ and p_1 may be taken as for the massive material, and so p_2 is obtained in terms of r_2 from the Kelvin equation, and hence the rate of transfer of material from kinetic theory. In this way, Shaler and Wulff[9] (1948) have found that the rate of transfer in copper at 850° C. from a flat surface to a concave surface of radius 10^{-2} cm. is such that the rate of change of the radius of curvature of the curved surface

Fig. 1

$\dfrac{dr_2}{dt}$ would be about 10^{-20} cm. sec.$^{-1}$, which means that an atomic layer would be added every 10^{13} sec., which is a very long time indeed. Recalculation of the problem by the present writer confirms that transfer of material (copper at 850° C., again) via the vapour phase cannot be appreciable, since even when r_2 is as small as 100 Angstrom units, an atomic layer is added only about every hour.

III—Surface diffusion in the first stage

There can be little doubt that *some* movement of material by surface diffusion occurs since it is known that the surface atoms of solids are highly mobile at temperatures near the melting point. However, the extents to which surface diffusion is responsible for the observed rates of first-stage shrinkage and sphering of pores remain unestablished.

The conclusions of Hüttig[10] (1942), from his extensive study of the change during sintering of many volume and surface properties, suggested that surface diffusion was mainly responsible for the early parts of sintering. Rhines[2] (1946) considered that surface diffusion might be, and Schwarzkopf[11] (1948) believed that it was, a mechanism in the first stage of sintering.

Shaler and Wulff[9] (1948) had the opinion that the role of surface diffusion in sphering of pores was small, and they stressed that data were lacking for a quantitative treatment similar to that which they had used for evaporation-condensation ; Shaler[1] (1949) believed that surface diffusion was probably important when the contact areas were small (i.e., early in the first stage).

Kuczynski[12] (1949) deduced that surface diffusion was the dominant mechanism in sintering of small particles (having diameter less than 30 microns), but more recent calculations by Cabrera[13] (1950) suggest that Kuczynski's deduction may not be valid. The work of Kuczynski and Cabrera will be discussed in the next section, so that their contributions to both surface and volume diffusion mechanisms may be taken together.

The present position is that the part played by surface diffusion in sintering is not yet known with any certainty.

IV—Volume diffusion in the first stage

Kuczynski[12] (1949) measured the rate of increase of contact area between a sphere and a plane surface of the same material. The contact area was circular and the value of n in the proportionality $x^n \propto t$ was determined, x being the radius of the contact area and t the time at a constant elevated temperature. n was used as an index of the dominant sintering mechanism occurring. Kuczynski adopted the treatment of Frenkel[14] (1945) showing that $n = 2$ for viscous flow and plastic flow, and deduced that $n = 3$ for evaporation-condensation. He also considered a possible *volume diffusion* mechanism whereby the surface forces tending to decrease the total surface area build a high concentration of vacant lattice sites in the material situated at and near the contact area between the sphere and plane ; the vacant sites then

diffuse away, giving rise to flow of material in the opposite direction—i.e., into the contact area. Kuczynski deduced that n would be 5 for this process. Again, if material were passing to the contact area by surface diffusion, n would be 7 according to Kuczynski. (But see Cabrera[13] (1950), referred to below.)

Kuczynski experimented with spheres and planes of copper, and later of silver. Since n was found to be approximately 5, it was concluded that *volume diffusion* was the dominant mechanism of growth of the contact areas. Kuczynski's theory of the volume diffusion mechanism enabled him to obtain values for the volume diffusion coefficient (D_v) from his experiments and, by evaluating D_v at a number of temperatures, he derived the heat of activation for volume diffusion (Q_v) and the constant D_v^o, where $D_v = D_v^o \exp.\left(\dfrac{-Q_v}{RT}\right)$, R being the universal gas constant and T the absolute temperature. The results for these sphere-on-plane experiments were as follows :—

Copper $Q_v = 56,000$ cal. mole^{-1} : $D_v^o = 70 \cdot 0$ cm.^{2} sec.$^{-1}$
Silver $Q_v = 42,000$ cal. mole^{-1} : $D_v^o = 0 \cdot 60$ cm.^{2} sec.$^{-1}$

They compare quite well with results of direct determinations using tracers. For example, the work of Johnson[15] (1941) gave :—

Silver $Q_v = 45,900$ cal. mole^{-1} : $D_v^o = 0 \cdot 89$ cm.^{2} sec.$^{-1}$

Dedrick and Gerds[16] (1949) studied the rate of growth of the contact area between adjacent spherical copper powder particles. Here again, an approximate fifth power relationship between the radius of the contact area and the time was found. Following Kuczynski, they concluded that volume diffusion was the operative mechanism. Their measurements gave :—

Copper $Q_v = 55,000$ cal. mole^{-1} : $D_v^o = 0 \cdot 12$ cm.^{2} sec.$^{-1}$

Kuczynski[17] (1949) later measured the sintering of silver wire to a silver block, again finding an approximate fifth power relationship and so concluding that volume diffusion was the mechanism. He obtained :—

Silver $Q_v = 45,000$ cal. mole^{-1} : $D_v^o = 1 \cdot 2$ cm.^{2} sec.$^{-1}$

Geach and Jones[18] (1949) made measurements of the growth of the initial line contacts between adjacent windings of copper wire on a bobbin. As their photomicrographs showed well, a cross-section of these windings bore a resemblance to an idealised section through a sintered mass of spherical particles. Their first results[19] indicated that $n \approx 5$ in the Kuczynski-type proportionality $x^n \propto t$ (x being the half-width of the contact area in this case) although the scatter of the experimental results threw some doubt on the validity of the figure. It was also found that $x^5 \propto a^2$, where $2a$ was the diameter of the wire, the experiments being conducted at constant temperature and time.

In the original sphere-on-plane work of Kuczynski[12], it was reported that the sintering of very small particles (<30 microns diameter) gave $n \approx 7$ and it was concluded that surface diffusion was the predominant mechanism.

We have now referred to all the experimental studies relevant to the Kuczynski approach (except for a study of glass sintering which is more appropriate to Part I, Section V.) It cannot be denied that the agreement among the results of the different sets of experiments and the quite good agreement of the Q_v and D_v^o values obtained with those from direct measurements are impressive.

We must now turn to criticism of Kuczynski's treatment of the volume and surface diffusion mechanisms.

In deriving his relation (equation (1) below) for a volume diffusion mechanism in the sphere-on-plane experiments, Kuczynski[12] employed a value for the concentration gradient of vacant sites in which it was implicit that a gradient existed only between the contact area and any point P (see Fig. 2) *just* outside it. It was not made clear what would happen to the excess concentration of vacancies which would be built up at P. It would probably have been more correct to have evaluated an average concentration gradient which must have existed between the contact area and the different parts of the sphere and block surfaces. (It is interesting to note, however, that the mechanism for the removal of vacant lattice sites postulated by Nabarro[20] (1948) might be applicable in this instance ; the vacancies would concentrate in planes within the lattice, the adjacent atomic layers then closing in with formation of dislocations.)

RADIUS AT BOTTOM OF CRACK = ρ
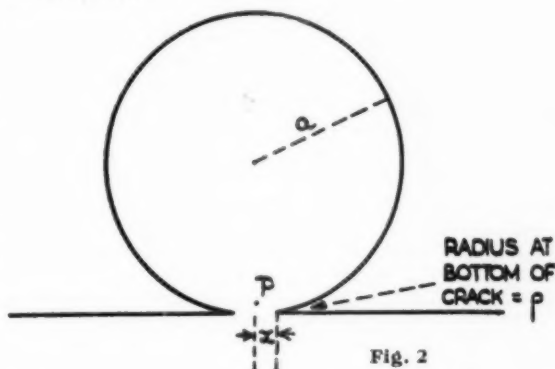
Fig. 2

Cabrera[13] (1950) recently gave a more precise treatment for volume diffusion and surface diffusion mechanisms in the sphere-on-plane experiments. The volume diffusion relations given by Kuczynski and Cabrera compare thus :—

Kuczynski
$$\frac{x^5}{a^2} = \left\{ 40 \cdot \frac{\sigma \delta^3}{kT} \cdot D_v \right\} t \qquad (1)$$

Cabrera
$$\frac{x^5}{a^2} = \left\{ 80 \, \pi \cdot \frac{\sigma \delta^3}{kT} \cdot D\delta^3 N\,(\infty) \cdot \frac{1}{\ln \dfrac{a}{\rho}} \right\} t \quad (2)$$

where
x is the radius of the contact area
a is the original radius of the sphere
σ is the surface tension
δ is the interatomic spacing
k is Boltzmann's constant
T is the absolute temperature
D_v is the coefficient of volume self-diffusion
t is the time at temperature T
D is the diffusion coefficient for vacant lattice sites
$N\,(\infty)$ is the equilibrium concentration of vacant lattice sites under a flat surface $[D\delta^3 N(\infty) = D_v]$
and ρ is the radius of the bottom of the " crack " formed between the sphere and plane (see Fig. 2).

It will be noted that equations (1) and (2) differ only in the " constant " term in curly brackets. The $x^5 \propto a^2$ proportionality for constant T and t which both equations give has been observed experimentally by Geach and Jones[19] (1950). In view of the different character of the

"constant" in equation (2) and in view also of the concentration gradient criticism referred to above, it is surprising that equation (1) has yielded such reasonable values of D_v and hence D_v^o.

The surface diffusion relations of Kuczynski and Cabrera compare as follow:—

Kuczynski
$$\frac{x^7}{a^3} = \left\{ 56 \cdot \frac{\sigma\delta^4}{kT} \cdot D_s \right\} t \tag{3}$$

Cabrera
$$\frac{x^5}{a^2} = \left\{ 160 \cdot \frac{\sigma\delta^4}{kT} \cdot (D_s)^{\frac{1}{2}} \cdot \delta^2 \cdot n(\infty) \cdot (\tau_s)^{-\frac{1}{2}} \right\} t \tag{4}$$

where

D_s is the coefficient of surface self-diffusion

$n(\infty)$ is the concentration of adsorbed atoms on a flat surface

and τ_s is a relaxation time.

Both Cabrera's equations (2), (4) give the same proportionalities $x^5 \propto t$ at constant T and $x^5 \propto a^2$ at constant T and t, meaning that volume and surface diffusion mechanisms would be distinguishable, not on these bases, but only by the differing heats of activation which might be involved. However, there are as yet few data for surface diffusion and one can merely say that the quite good agreement between the heat of activation values obtained from the Kuczynski-type experiments and the results of *volume* diffusion tracer determinations may be significant.

Although Cabrera does not claim validity of his calculations for small particles, his work suggests that Kuczynski's experimentally determined proportionality $x^7 \propto t$ (for spheres less than 30 microns diameter) may not be correctly explained by surface diffusion. Further doubt arises since Kuczynski[12] found that the heat of activation for the sintering of these smaller spheres was 56,000 cal. mole[-1], the same as for the larger spheres, and present indications are still that the larger spheres sintered by a *volume* diffusion mechanism.

V—Viscous and plastic flow in the first stage

Frenkel[14] (1945) supposed that crystalline bodies could exhibit a Newtonian viscosity at high temperatures. (Nabarro[20] (1948) has since shown that crystalline materials may only be expected to flow viscously at an appreciable rate if a mosaic structure be assumed. However, this does not invalidate the work referred to below.) Frenkel then proceeded to derive an expression for the rate of coalescence of two equal spheres initially in contact at a point. The derivation involved the assumption that, as the contact changed from a point to circles of increasing radius, the parts A', B' (see Figs. 3a and 3b) would remain portions of spheres. The change of total surface energy was equated to the work done in the flow of material (obtained from viscosity theory), giving the result that the rate of change of contact area was constant (i.e., $\theta^2 \propto$ time t).

Bernhardt and Shaler[1] (1949) plotted θ^2 against t for copper spheres and drew a curve as indicated in Fig. 4, but, if all the experimental data were included on the graph published, there seems to be little justification for drawing the straight portion II. However, they believed that the curve was explained as follows:—When the area of contact was small, the attractive forces between the spheres (an estimation of which was attempted, as mentioned in Part I, Section I) would be so great that there would be immediate *plastic* flow which would continue until the stress fell below the critical value for it, at which time *viscous* flow under lower stresses would occur. It was suggested that the plastic flow was
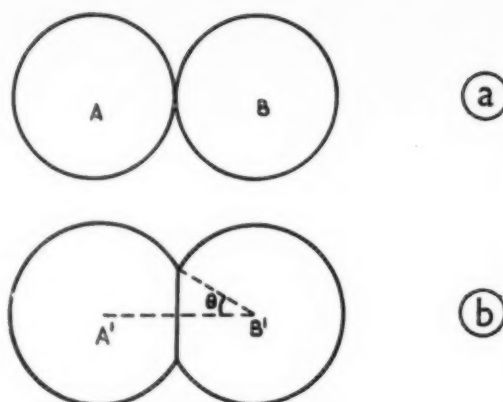
Fig. 3 (a and b)

responsible for the initial rapid coalescence manifested by portion I of the θ^2/t curve (Fig. 4), while the slower viscous flow gave portion II. The linear portion was said to begin at about 5,000 lb. in.[-2] at 800° C. and a few hundred lb. in.[-2] at 950° C., but these stress figures are suspect, since they are based on the calculation of the attractive force between copper surfaces. Shaler suggested that portion II of the curve represented the second stage of sintering, but, according to our definitions of the two stages, this cannot be, since the transition from portion I of the curve to portion II occurs when $\theta^2 \approx 0.01$ radian[2] on the 850° C. curve, giving $\theta \approx 6°$. This is hardly likely to have given isolated pores.

Kuczynski[21] (1949) observed that a glass sphere sintered to a glass plane according to the proportionality $x^2 \propto t$ (x being the radius of the contact area at time t, temperature constant), a result in agreement with Frenkel's relation for *viscous* flow. The calculated values of the viscosity coefficient were given as ranging from 1.27×10^{12} poises at 575° C. to 7.00×10^7 poises at 743° C., but these figures are π times too large. (This particular paper contains several errors, some typographical. It may conveniently be mentioned here that no attempt has been made to point out errors in the papers quoted throughout this review unless the errors were considered to be of consequence). The heat of activation for the sintering varied from 121,000 cal. mole[-1] for temperatures below about 700° C. to 67,000 cal. mole[-1] for temperatures above. This sort of variation was also found by Clark and White (see
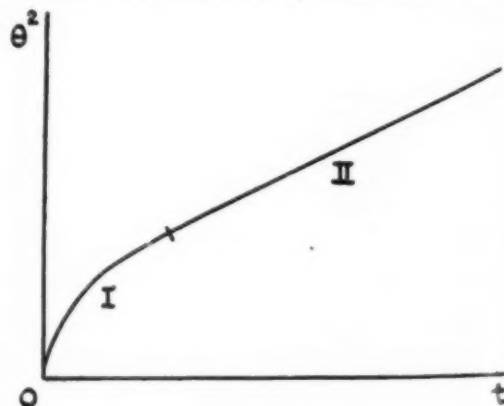
Fig. 4

below) in their glass sintering studies and they pointed out that it was not discordant with the known properties of glass flow.

Clark and White[22] (1950), at Sheffield University, studied sintering of glass, alumina and magnesia as a chemical rate process* with the object of determining the heat of activation associated with the flow of these materials. The rate of shrinkage of pressed compacts in air was measured.

Shrinkage curves were also obtained theoretically on the following basis. It was assumed that equal spherical powder particles, initially in contact at a point, sinter together with formation of a lens L between them, the portions A' and B' remaining spherical (Figs. 5a and 5b). The material in L is derived from A, B and flows into L under the influence of surface tension forces. It was further assumed that the flow is confined essentially to the surface layers of the spheres (note the divergence from Frenkel's treatment). Both *viscous* (Newtonian) and Bingham *plastic* flow were considered. (In Newtonian flow, the rate of shear strain is proportional to the shear stress, the constant of proportionality being the
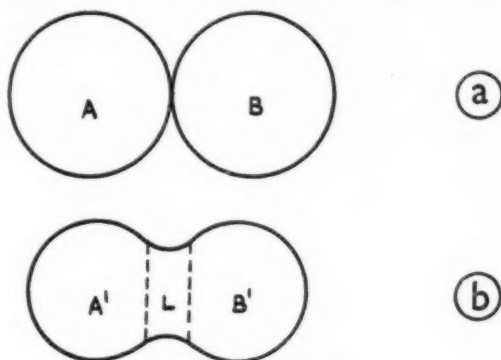

Fig. 5 (a and b)

reciprocal of the viscosity coefficient. With Bingham flow, the rate of shear strain is proportional to the difference between the shear stress and a critical shear stress). In each case, the rate of shrinkage of a compact of such spherical particles was expressed in terms of a number of factors, including a velocity constant which was related to the appropriate viscosity coefficient.

In order to fit the theoretical curves to the experimental ones, an empirical adjustment of the rate of shrinkage equation became necessary. This was considered to be due to angularity of the powder particles used. Experiments were conducted at a number of different temperatures and a plot of the logarithm of the velocity constant against the reciprocal of the absolute temperature (the Arrhenius relation) gave the heat of activation for the flow. It was found that a soda-lime glass sintered with a *Newtonian viscosity*, with heat of activation varying from approximately 75,000 cal. mole⁻¹ at 676° C. to 150,000 cal. mole⁻¹ at 598° C. Both α–alumina and magnesia sintered according to the *Bingham* law. The alumina flow had constant heat of activation 87,000 cal. mole⁻¹. The heat of activation of the magnesia flow was uncertain.

The simultaneous assumption that flow into the lenses is confined to surface layers and that the particles remain spherical (which involves a volume type of flow) during

* See also Part I, Section VI on this subject.

this process is open to criticism, as also is the empirical adjustment found necessary in the final shrinkage rate equation. The latter objection may be removed when spherical powder particles are used. (Such particles of alumina have been prepared by Roberts, Carruthers and Plummer[23] (1949), of Leeds University, who are using them in a programme of shrinkage studies, as yet unpublished). Clark and White emphasised that the theoretical treatment was merely a device to allow evaluation of the velocity constant, and they did not claim authenticity for the mechanism postulated. The heat of activation results for glass were similar to those given by Seddon[24] (1939) and to results[25] obtained by applying the treatment of Mackenzie and Shuttleworth (see Part II, Section II) to the experimental data of Clark and White.

Clark and White[25] found that their theoretical curves agreed with the experimental ones, not only when the pores were connected (our first stage of sintering), but also after they became isolated (second stage); in the latter case the entrapped air must have had a negligible effect.

Plastic flow mechanisms for the growth of contacts were previously suggested by Rhines[2] (1946) and Schwarzkopf[11] (1948).

Mackenzie and Shuttleworth[26] (1949) pointed out that differential flow, by which it is assumed they meant *viscous or plastic flow*, might be the dominant mechanism in the first stage of sintering. Considering a void space in a compact, the inward pressure due to surface tension would be greatest in the neighbourhood of those parts of the surface of the void having the greatest concavity, giving the greatest flow there, and hence growth of contact areas and sphering of the pores. Commenting on the system of a sphere on a plane (cf. Kuczynski[12]), Mackenzie and Shuttleworth emphasised that the shear stress would be practically zero everywhere except at the contact, so that the bulk of the sphere could not move *plastically*, giving a constraint which would effectively prevent differential flow occurring. (They believed *volume diffusion* to be the dominant mechanism in Kuczynski's work, in agreement with Kuczynski's view for the larger spheres).

VI—Sintering as a rate process

Data on shrinkage during sintering have been examined by a number of workers with the specific purpose of determining whether they can be correctly interpreted in terms of a rate process, as met in chemical reactions. Although the approach is, in principle, applicable to both stages of sintering, the work published up to the present has dealt mainly with compacts whose densities suggest that the pores were connected, and so it is convenient to mention the researches now.

A characteristic of a rate process is that, if time t_1 is taken to pass from one given state to a second given state at absolute temperature T_1, and time t_2 for the same change at temperature T_2, then

$$\ln\left(\frac{t_1}{t_2}\right) = \frac{Q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \quad \text{(after Arrhenius)} \quad (5)$$

where R is the universal gas constant

and Q is the heat of activation for the process.

Hüttig[27] (1948) first applied this relation directly to sintering, using data obtained for iron powders by Bernstorff[28] (1948). The non-preheated compressed powder was taken as the first state and a particular

degree of shrinkage as the second state. One powder gave a straight line plot of $\ln t$ against $\frac{1}{T}$ for higher temperatures when the second state was well advanced; the Q value obtained was 51,000 cal. mole^{-1}, which, according to Hüttig, compares with 58,000 cal. mole^{-1} derived for the heat of activation for volume self-diffusion of iron by van Liempt using an approximate method. Unfortunately, the other data did not support the first Q value, so the result is not convincing.

Jordan and Duwez[29] (1949), in an impressive programme of experimental work, measured the shrinkage rates of copper compacts *in vacuo* and in hydrogen, and applied the above relation (5). Both sets of experiments gave *approximate* straight line plots, the Q values being 128,000 cal. mole^{-1} and 80,000 cal. mole^{-1} respectively; it was suggested that the difference might be due to removal of oxide films on the particles by the hydrogen. The Q values are very large compared with values of the heat of activation for volume self-diffusion of copper derived from tracer methods by different workers (these range from 44,000 to 61,000 cal. mole^{-1}).

The plotted points of Jordan and Duwez actually showed a systematic slight deviation from the straight lines drawn for determination of the above Q values. This led to the suggestion that sintering may be explained in terms of a number of rate processes, one of which is predominant. The standard states adopted give little doubt that the results applied to our first stage of sintering. Extension of the work to near complete sintering would be of considerable interest as it would show clearly whether different heats of activation are associated with the two sintering stages.

The work of Clark and White (1950) was described in the previous Section. Their approach was complicated because they were also interested in viscous and plastic flow mechanisms of sintering, and they desired to evaluate viscosities. However, the Arrhenius type of relation was applied. The sintering of alumina gave a good straight line plot of the type $\ln t$ against $\frac{1}{T}$ Q being 87,000 cal. mole^{-1}. Sintering of a soda-lime glass, on the other hand, gave a curve and hence a variable Q, as stated in Part I, Section V. The suggestion of a superposition of different rate processes applies in this case also.

The work mentioned in this section was done specifically to test the validity of the Arrhenius relation in sintering, but, as the reader will be aware, the use of this form of relation is of course widespread.

VII—Retrospect on the first stage

There remains little doubt that an *evaporation-condensation* mechanism in the first stage of sintering can make only a negligible contribution to the observed phenomena. The role of *surface diffusion* is still uncertain. (These remarks apply also to sphering of pores in the second stage.)

Present indications are that a *volume diffusion* mechanism is operative in the variety of Kuczynski-type experiments which have been done, except in the case of glass. The work of Kuczynski, and Clark and White point to a *viscous flow* mechanism in glass-sintering. Impressive evidence in favour of a *plastic flow* mechanism in the first stage is produced only by Clark and White; further work is required before the role of plastic flow can be assessed with certainty.

PART II—SECOND STAGE OF SINTERING—ISOLATED PORES
I—General

In all discussions of the second stage, we shall be concerned with evacuated pores unless otherwise stated.

When the void space in the compact is confined to isolated pores, a state of affairs which is bound to result from continued growth of the contact areas between particles, no change in density but only sphering can be caused by movement of material via the *vapour phase* or by *surface diffusion*. Pines[30] (1946), Ivensen[31] (1947) and Shaler[1] (1949) reached this conclusion.

Also, no change in density can be caused by a *volume diffusion* mechanism; unless void volume passes through the exterior surface of the compact; for this mechanism, one might imagine an increase of density resulting from the passage of vacant lattice sites from the pores to the exterior surface of the compact. Pines[32] (1946) gave a mathematical treatment for this. Such diffusion is a slow process* and large distances would have to be traversed by the vacant sites. Further, as Mackenzie and Shuttleworth[26] point out, no net diffusion of vacant sites from a given point in the compact to the exterior surface could occur until there were no pores between the point and the exterior surface. The mechanism cannot be operating to any marked extent as it would lead to sintering from the exterior surface inwards and the time of sintering would be a function of shape and size of compact.

The conclusions of Hüttig[10] (1942) on the mechanism of sintering largely involved surface and volume diffusion, and Rhines[2] (1946) believed that volume diffusion was predominant. However, for the reasons outlined above, these ideas are not acceptable as explanations of the *change in density* during the second stage.

Viscous flow and *plastic flow* remain possible mechanisms of the density change. We will now consider the work giving evidence for them.

II—Viscous and plastic flow in the second stage

If the solid material in a compact with isolated pores be assumed incompressible, the surface tension forces acting in the pore surfaces are equivalent to a hydrostatic pressure acting on the external surfaces of the compact. Such pressure produces an equal uniform hydrostatic pressure at all points throughout the whole volume, so that, on the basis of a viscous or plastic flow mechanism, the rate of increase in density will be uniform throughout the whole body, and the rate of sintering will be independent of shape and size, as is actually observed. This is an important point in favour of the hypotheses that viscous or plastic flow mechanisms are acting. It has been discussed by Mackenzie and Shuttleworth[26] (1949).

Shaler, Wulff and Udin have done three illuminating sets of experiments, as follow :—

(i) Shaler and Wulff[9] (1948) measured the rate of shrinkage *in vacuo* of compacts of spherical copper particles at sintering temperatures.

(ii) Shaler[1] (1949) measured the rate of shrinkage at 1,000° C. of evacuated single cylindrical pores, made by drilling holes longitudinally in copper wire and stopping the ends of the holes with copper plugs.

* Mackenzie and Shuttleworth[26] (1949) state that, if the excess concentration of vacant lattice sites in copper at 1,000° C. is produced by pores of 1μ radius, and diffusion has to take place over a path of 1 mm., then the decrease of linear dimensions due to volume diffusion will be only 3×10^{-8} cm.day^{-1}.

(iii) Udin, Shaler and Wulff[33] (1949) studied the flow of copper wires subjected to low stresses at temperatures near the melting point and found it to be viscous. These experiments will be constantly referred to as we develop discussion of theoretical approaches.

Most of the work relevant to this section is closely interwoven, but it will be convenient to deal with *single* pores first and then pass on to *multi-pore* systems.

Frenkel[14] (1945) considered theoretically the closing of a *single* spherical cavity in an infinite homogeneous viscous medium. The treatment was extended by Shaler[1] (1949) (who incorporated a correction of Frenkel's work, due to Eshelby[1a] (1949)) for the closing of a cylindrical pore by viscous flow and applied to the experiments (ii) cited above. It was suggested that there was agreement between experiment and theory when the values of surface tension and viscosity derived from the wire-pulling experiments (iii) were adopted, and Shaler concluded that the pores shrank by viscous flow. However, scrutiny of the published experimental and theoretical plots of shrinkage of the single pores against time shows that the agreement is not good, throwing doubt on the validity of Shaler's conclusion.

Turning now to *multi-pore* systems, a further extension of Frenkel's treatment for closing of a single pore was made by Shaler and Wulff[9] (1948) to explain the results of the compact experiments (i). In their model, the solid material which surrounded Frenkel's pore was replaced by porous material with the intention of deriving an expression for the rate of shrinkage of a compact containing many equal closed pores. However, as pointed out by Shuttleworth[1a] (1949), the calculations do not allow for the fact that all the pores in the compact close simultaneously, and so they must be discounted.

Mackenzie and Shuttleworth[26] (1949) obtained an expression for the rate of shrinkage of a compact containing equal spherical pores by viscous flow, using a model originally indicated by Fröhlich and Sack[34] (1946). A pore of radius r_1 was surrounded by a spherical shell of incompressible solid material out to radius r_2, which was of such magnitude that the pore + shell had the same density as the whole compact. The solid material and pores outside the shell were replaced by an equivalent homogeneous continuum. Shrinkage of the compact could then be considered in terms of the rate of change of the dimensions of the pore + shell when pressure $-\dfrac{2\sigma}{r_1}$ was applied inside the pore (σ being the surface tension). Assuming viscous flow of the solid material, the energy dissipated in the flow was equated to the change in surface energy, giving a differential equation for the density as a function of time. Application of the solution of this equation to experimental data on shrinkage of compacts would allow derivation of the ratio of the surface tension σ to the viscosity η. Mackenzie and Shuttleworth also considered the effect of gas in the pores of a viscous solid during sintering.

It is to be expected that the viscous solid model will explain the sintering of glass ; full confirmation of this view may emerge from the work of Clark and White (see below). When applied to the results of Shaler and Wulff's copper compact experiments (i), Mackenzie and Shuttleworth's viscous flow treatment gave $\eta = 2 \cdot 2 \times 10^9$ poises at 850° C. This value compared unfavourably with $\eta = 7 \cdot 8 \times 10^{13}$ poises which was derivable from Udin's wire-pulling experiments (iii) and with $\eta = 3 \cdot 4 \times 10^{12}$ poises given by Nabarro's[20] theory of vacant lattice site diffusion (both at temperature 850°C.).

Mackenzie and Shuttleworth concluded that the sintering of copper could not be explained by *viscous* flow, and they suggested that the important mechanism for deformation during sintering occurred when the yield stress was exceeded—i.e., the flow was *plastic*. A plastic flow mechanism had been previously suggested by Jones[35] (1937), Bangham[3] (1947) and Schwarzkopf[11] (1948).

In support of their view, Mackenzie and Shuttleworth pointed firstly to the viscosity discrepancy mentioned above. Secondly, they emphasised that the rate of closing of pores in the copper compact experiments (i) was much greater than the rate of change of the dimensions of the single pores in experiments (ii) and of the wires in experiments (iii). Now in the neighbourhood of a single pore the shear stress caused by surface tension decreases with distance from the centre of the pore, so that, if flow occurs only when a critical stress is exceeded, the material beyond a certain distance corresponding to the critical stress will not be able to flow. In this case, no flow can occur to enable the pore to shrink. On the other hand, in a porous compact the plastic regions surrounding each pore will overlap and flow will occur because the yield stress is exceeded everywhere. Thus, the concept of a plastic flow is attractive for an explanation of the differing behaviour of single pores and of pores in compacts. Thirdly, Mackenzie and Shuttleworth suggested that the mechanical properties of copper at high temperatures are similar to those of tin at room temperature observed by Chalmers[36] (1936). The tin exhibited viscous flow (" microcreep ") with $\eta = 10^{15}$ poises at low stresses, but when the stress exceeded the critical value of 10^7 dyne. cm.$^{-2}$ a much greater rate of deformation occurred. Udin's viscous wire-pulling experiments (iii) under low stresses appear to correspond to the microcreep of tin, and the rate of deformation necessary to explain sintering of copper appears to correspond to the flow of tin when the critical stress is exceeded.

Using the same model and procedure as for their viscous treatment, Mackenzie and Shuttleworth arrived at an expression for the rate of shrinkage of a compact on the basis of Bingham plastic flow (the rate of shear strain is proportional to the difference between the shear stress and a critical shear stress). Making the assumption that copper behaves like a Bingham solid at high temperatures, Mackenzie and Shuttleworth applied their treatment to the results of the experiments (i), (ii) and (iii), and obtained a rough estimate of the yield stress τ_e. In the wire-pulling experiments (iii) of Udin, Shaler and Wulff, yield had not occurred, giving $\tau_e > 2 \cdot 0 \times 10^5$ dyne cm.$^{-2}$ at 1,000° C. Rapid sintering occurred in Shaler and Wulff's experiments (i), suggesting that the yield stress was exceeded and giving the result that τ_e might be greater than $2 \cdot 6 \times 10^5$ dyne cm.$^{-2}$ at 850° C. In Shaler's single pore experiments (ii), the pores did not close appreciably when heated to 1,000° C., giving $\tau_e > 0 \cdot 2 \times 10^5$ dyne cm.$^{-2}$. The only evidence suggesting that these indications of the value of τ_e are of the correct order is that the critical shear stress of copper single crystals is 10^7 dyne cm.$^{-2}$ at room temperature.

The theoretical work of Mackenzie and Shuttleworth awaits more data. The authors stressed the need for experimental determination of (i) the laws of deformation of materials under small stresses at sintering

temperatures, (ii) the density of a compact of equal spheres *in vacuo* expressed as a function of the pore size, time and temperature, and (iii) the effect of small external pressures on sintering. Preliminary experiments along these lines are being undertaken by Geach and Jones[19] at the Associated Electrical Industries Research Laboratory, Aldermaston.

As mentioned in Part I, Section V, when the work of Clark and White was discussed, it was found that their theoretical treatment, which was derived for the first stage of sintering, gave shrinkage curves which fitted experimental curves (sintering in air) relating to *both* the first and second stages[25]. (Their results indicated that a soda-lime glass sintered with Newtonian viscosity and that α-alumina and magnesia sintered by Bingham plastic flow ; see Part I, Section V for other details.) It is noteworthy that Clark and White have also applied the viscous flow treatment of Mackenzie and Shuttleworth (which dealt with the second stage) to their experimental data for shrinkage of glass compacts (the data referred to both stages) and have obtained the same value for the heat of activation as given by their own treatment[25].

These two examples of successful application of a treatment for one stage of sintering to the other stage are evidence suggesting that viscous or plastic flow may be the predominant mechanism in both stages, in which case the division of the subject into two loses its usefulness. (It will be remembered that the division was made on the grounds that there were more possible mechanisms for shrinkage in the first stage than in the second, which suggested that shrinkage studies might have to be considered in two parts.)

III—Retrospect on the second stage

We are concerned with shrinkage of a system of closed pores. Of the three possible mechanisms, it has been shown that *volume diffusion* could not account for observed shrinkage rates. It becomes a case of determining the applicability of *viscous and plastic flow* mechanisms.

Attention has been focused mainly on copper and glass. The arguments of Mackenzie and Shuttleworth in favour of a predominant plastic flow mechanism in

second stage sintering of copper are plausible, but experimental support for the Bingham flow assumption is lacking as yet. All present indications are that second stage sintering of glass occurs by viscous flow.

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Gas Turbine School Re-Opens

BRITAIN'S School of Gas Turbine Technology—the only one of its kind in the world—is re-opening at Farnborough Place, Farnborough, Hampshire, in October. The School, maintained by Power Jets (Research & Development), Limited, has been moved because of expansion from its old premises at Lutterworth, where Air Commodore Sir Frank Whittle's team designed and tested some of the earliest jet engines.

Founded in 1944 to instruct Dominions Air Force personnel in aircraft jet engines, the School now gives instruction in the technicalities of the use of gas turbines in high speed aeroplanes, locomotives, road vehicles, ships, power stations, process power and other industrial applications. The School has a wide range of gas turbine equipment and engines, including test houses and a jet aircraft on which installation and practical ground instruction is given. On every course actual test-bed runs on shaft-power and jet engines are undertaken by the students themselves.

There will be four different types of courses, one of which is an International course designed specially for overseas engineers. The first International course at Farnborough will be held before the end of the year.

I.M.M. Abstracts

THE Institution of Mining and Metallurgy has commenced publication of monthly abstracts of world literature on economic geology and mining (excluding coal), mineral dressing, extraction metallurgy (excluding iron but including refining), and allied subjects of interest. Annual subject, and author indexes will be issued. For the time being *I. M. M. Abstracts* will be published as part of the *Bulletin* of the Institution and will be issued free to Members and Subscribers. The subscription rate for the *Bulletin*, including I. M. M. Abstracts, is £3 per annum (12 monthly issues) and enquiries should be addressed to the Institution at Salisbury House, Finsbury Circus, London, E.C.2.

Soldering of Aluminium

Avoidance of Brittlement

A communication from the Aluminium Development Association

More than twenty years ago a series of papers was presented to the Institute of Metals on the penetration of metals by molten solders. With the increasing application of soldering to aluminium alloys the question again arises, and in this article, the Association presents the conclusions reached as a result of an investigation of this aspect of the problem.

ALTHOUGH welding and brazing give excellent results when applied to the joining of aluminium and its alloys, there are certain applications in which the relatively high operating temperatures are disadvantageous, especially in electrical work where there would be some danger of damaging the insulation. For such purposes, soldering is often preferable : thus, the jointing of electric cable could hardly be effected by any other means. The soldering of aluminium presents certain difficulties caused primarily by the presence of oxide film, and the method recently announced[1] for producing wiped joints on aluminium-sheathed cable represents an important technical advance.

This method, derived from a process developed by The British Aluminium Co., Ltd.[2] begins with the removal of the oxide film by filing. This is particularly suited for site work, but in the workshop a mixture of phosphoric and nitric acids may be used ; this removes the bulk of the oxide and leaves the rest in a readily abradable condition. After this abrasive or chemical treatment, the metal is " tinned " by melting on to the surface a 90/10 tin-zinc alloy with a blowlamp. To promote wetting, the film of molten tinning metal is brushed with a wire brush and the excess finally removed. In another method now being developed, the oxide film is dispersed by supersonic energy applied to the tinning metal.

The aluminium surface thus coated with tinning metal is readily soldered, and the wiped joints common in cable practice can be made without difficulty using the normal 50/50 tin-lead solder. The joint is then finished with a protective layer of resin or grease.

This process is simple, differing only slightly from conventional methods, and has been shown by accelerated " life " tests to give durable joints. It constitutes an important advance, and its application to other fields may be expected. It is therefore important to appreciate the conditions under which the process works to the best advantage.

Solder Embrittlement

Experience with other metals indicates that when a metal or alloy is in contact with a liquid metal or alloy of lower melting point, there is a possibility that penetration may occur, the liquid attacking the solid metal, particularly at the grain boundaries, and causing a degree of weakness and embrittlement. Thus, in 1927, a series of papers[3] was presented to the Institute of Metals showing that brass and copper may be penetrated by tin and solder and that mild steel can be penetrated by brazing solder. Later, Wesley Austin[4] studied the behaviour of a variety of steels and non-ferrous metals

and alloys when subjected to the joint effects of stress and molten tin and solder, concluding that " almost all the usual metals, alloys and steels, employed in general engineering, if tensile stressed and in contact with molten solder, are susceptible to penetration and weakening to a greater or lesser degree." Cracking under stress may occur in the elastic range and cold-worked material is generally more subject to penetration and embrittlement than annealed.

Tinning Under Tension

Tests have been made to ascertain whether the new processes for soldering aluminium are liable to cause embrittlement of this kind ; the experimental method used, and the results obtained are detailed below.

In the investigation, 2 in. gauge length tensile test pieces were first swabbed for 2 minutes with a solution containing 20% phosphoric acid and 10% nitric acid, with a trace of a wetting agent. They were then extended to known degrees in a Hounsfield tensometer and held at that extension whilst being tinned. The strain includes both elastic and plastic deformation, but the elastic correction will be small, particularly for the work-hardening materials such as pure aluminium and the aluminium-manganese and aluminium-magnesium alloys. Efforts were made to determine the amount of strain which can be allowed without undue penetration, performance being judged by whether or not the test pieces fractured during tinning or during subsequent cooling to room temperature.

A 1 in. length of the test pieces was tinned with 90/10 tin-zinc alloy using a 125 W. electric soldering iron whose temperature was set at 300° C.* In no case did tinning take more than 1 minute. No difference was observed between the critical strains for specimens taken in the longitudinal and transverse directions in any of the materials tested. Pure aluminium, aluminium-manganese alloy (soft or half hard), or annealed aluminium-magnesium-silicon alloy (HS. 10–0, B.S. 1470) samples did not fail by cracking when strained as far as possible under the conditions of this test : there is some possibility, however, that they would have failed had the heat been applied for a longer time : that is, had solder penetration proceeded further.

Of the susceptible materials, fully heat-treated aluminium - magnesium - silicon alloy (HS. 10–WP, B.S. 1470) and a fully heat - treated aluminium - magnesium-zinc alloy were by far the worst, failure occurring at an early stage in tinning and often before the material had been fully softened by the heat absorbed from the iron.

In the case of the aluminium-magnesium alloys such rapid failures only occurred with large strains :

1 See, for example, *Light Metals*, March, 1949, p. 130.
2 B.P. 634,823 (The British Aluminium Co., Ltd.).
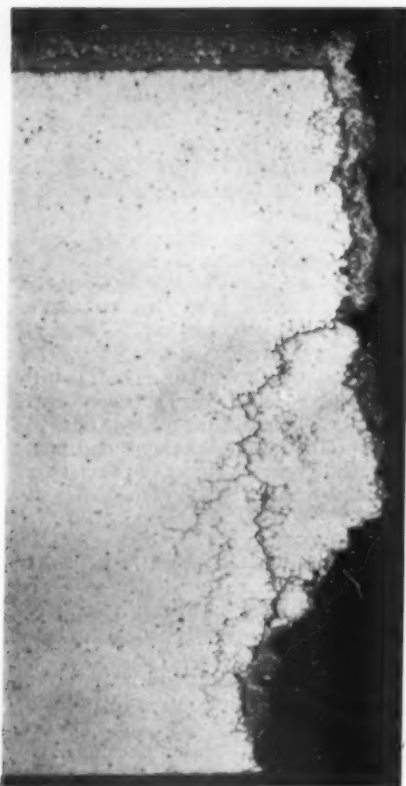3 H. J. Miller, *J.Inst. Metals*, 1927, **37**, 183.
   H. J. Gurtley, *ibid*, p. 193.
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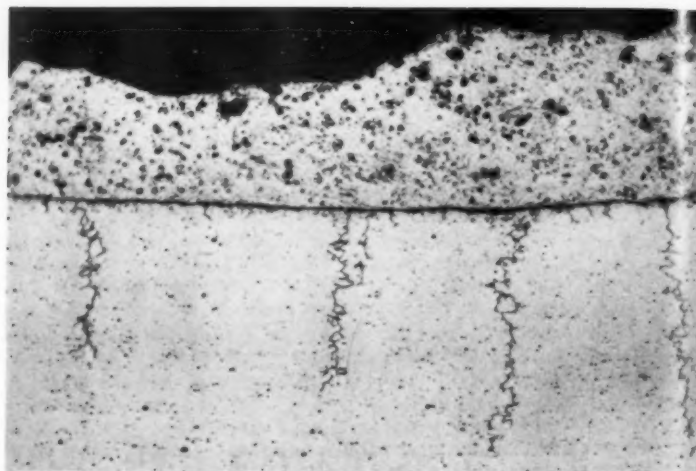4 G. Wesley Austin, *J.Inst. Metals*, 1936, **58**, 173.

* On touching the specimens, however, the temperature of the bit fell to about 250° C.

Cracks at the base of Erichsen impressions in soft aluminium—3% magnesium alloy sheet after filling the impression with solder × 100

Crack in automobile body component in aluminium—3% magnesium alloy soldered with 70/30 tin-zinc solder. The crack has completely penetrated the section, which had been work-hardened at this point by a forming operation. × 100

with strains around the critical value, failure often occurred after the iron had been removed. For the aluminium-magnesium alloys (NS. 4 and 5), solder cracking occurs in material with more than about 4% strain using the 90/10 tin-zinc solder, the critical value for NS. 5 being a little lower than for NS. 4.

Another Experimental Method

Because of the possible use of soldering as a means of filling dents in sheet—e.g., automobile body panels, studies have been made on the occurrence of brittleness when carefully made impressions in aluminium-alloy sheet were filled with solder. The impressions were made on soft 18 S.W.G. NS. 5 strip and indentations with an Erichsen machine so that the impressions were 3, 4, 5, 6, 7 and 8 mm. deep. The indented surfaces were tinned with a 70/30 tin-zinc alloy. No cracks appeared immediately, but when the specimens were examined the following day, intercrystalline cracking was often found : this was some 16 or 17 hours after soldering. It is thus apparent that on work-hardened strip, cracking can be produced by the tin-zinc solders. Other tests using a similar method have shown that pure aluminium and the aluminium-manganese alloy do not undergo cracking during soldering whether annealed or work-hardened and that the aluminium-magnesium alloys (NS. 4 and NS. 5) are susceptible to cracking only when work-hardened. Thus the results given by the dent-filling tests are consistent with those obtained under tension. Since the rate of penetration is likely to be influenced by the temperature, soldering should be carried out at as low a temperature as possible, and

freedom from cracking may be ensured by preliminary annealing of the deformed area in the neighbourhood of the dent which is to be filled.

Apart from the above methods of avoiding solder cracking in susceptible material such as the aluminium-magnesium series, there remains the possibility of using a solder which will not cause cracking. The melting point of pure zinc is above the temperature required to anneal aluminium alloys, so that a solder based on zinc is unlikely to produce cracking after short heating because of the annealing effect. This conclusion has been checked experimentally. A number of other possible materials have been tried as solders, but apart from pure tin, only a 95/5 tin-cadmium solder gives results superior to those obtained with the 90/10 tin-zinc alloy. The improvement was not, however, considered sufficient to remove the danger of solder cracking in NS. 5 alloy and it seems that the only practical method of ensuring freedom from cracking in the filling of dents is to anneal beforehand.

Conclusions

The experimental work just described has shown that, in common with most other metals, some aluminium alloys may be subject to brittleness and cracking as a result of penetration by solder during tinning. This does not affect all aluminium alloys and, in particular, pure aluminium is free from any tendency to the defect. The same consideration also applies to the aluminium-manganese alloy (NS. 3 in B.S. 1470) and to the aluminium-magnesium alloys NS. 4 and 5 when annealed.

The method of soldering which has been described, involving pre-treatment by abrasion or by phosphoric-nitric acid treatment followed by tinning with a tin-zinc solder and finishing with tin-lead, may be considered a perfectly practical workshop method for many applications. For many purposes, where durability of the joint under immersed conditions is required, and protection impracticable, gas welding and brazing will remain the principal methods of joining. The closely similar chemical nature of the parent plate and the joining

metal is naturally an advantage when the joints are to be subjected to corrosive conditions. On the other hand, there are applications where the low temperatures experienced in soldering are advantageous. For such purposes, provided that account is taken of the necessity for annealing certain of the alloys if they have been locally cold worked, there is every reason to expect satisfactory service when the abrasive soldering method is used.

Methods of Conveying in Electric Furnaces

By James McDonald
McDonald Furnaces Ltd.

During the last 10–15 years, little fundamental development has taken place in the means of conveying the charge through continuous-type electric heat-treatment furnaces. A number of difficulties have been experienced with the various methods and the present phase of development appears to be one of consolidation based on the scientific exploitation of existing methods.

SURVEYING, generally, the present-day design of electric furnaces for preheating and heat treatment work, there seems to have been little fundamental departure from the acknowledged methods of conveying existing 10 to 15 years ago. It is significant that, adhering to old ideas, particularly in the face of increased competition, difficulty is being experienced in producing new designs with higher furnace efficiencies. It is apparent that the furnace manufacturers are, unknowingly perhaps, passing through a phase of consolidation, and are scientifically exploiting every past development.

Emerging from this policy, we have three types of continuous furnace, which have been re-designed or modified for adaption to new applications. They may be classified as follows :—

(1) Walking Beam Furnaces.
(2) Flash Annealing Wire Rope Furnaces.
(3) Shaker Hearth Furnaces.

Walking Beam Furnaces

The first type is well known to the gas- and fuel-fired furnace engineers, but by judicious design, necessitating meticulous care with the hearth seals, to prevent the ingress of air, this type of furnace is now being used successfully with electricity as a heating medium.

Ingots, plates, etc., can be handled through a walking beam furnace without the aid of non-productive conveyor weight and, consequently, the consumption per ton is relatively low. It is interesting to note that this design of furnace is rapidly gaining popularity, but that it does not incorporate a true walking beam system.
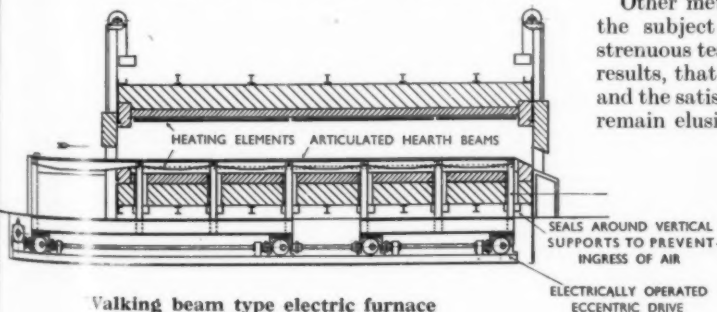
In annealing processes for steel sheets, etc., it is important that a sheet is conveyed without the risk of scratching, which requires a design of mechanism to ensure a vertical lift and a horizontal travel. For the preheating of ingots and plates for pressing, this feature is not essential and the beams may be operated by a simple eccentric motion.

Flash Annealing Wire Rope Furnaces

Flash annealing of aluminium alloy sheets and discs by forced convection is a comparatively new process, whereby annealing can be completed in a few minutes. Obviously, conveyor weight must be restricted, and the American idea of using stainless steel wire ropes has been introduced with a certain degree of success. Finished sheets and discs must be finally annealed without showing any traces of scratching or marking and this requirement has presented a serious problem to both the furnace manufacturers and users. All types of rope constructions have been tested, without obtaining complete satisfaction. The matter is of considerable importance, and a supreme effort is being made by some manufacturers to use asbestos, either as an inherent part of the rope or as a sheath. Whilst no marking is experienced by this method, the life is problematical, and the idea can only be justified after sufficient experience has been gained, to ensure that production is not interrupted unnecessarily. To facilitate replacement of the new ropes, numerous types of quick release clips have been introduced, but opinions differ considerably as to their success.

Other methods of conveying, which will probably be the subject matter of patents, are now undergoing strenuous tests, and it is more than likely, from the early results, that steel wire ropes will eventually be replaced and the satisfactory answer to the problem will no longer remain elusive.

Shaker Hearth Furnaces

It would appear that furnace manufacturers are again marketing the reciprocating tray furnace, now generally referred to as a shaker hearth furnace. Furnace designers who have been engaged on this

HEATING ELEMENTS   ARTICULATED HEARTH BEAMS

SEALS AROUND VERTICAL SUPPORTS TO PREVENT INGRESS OF AIR

ELECTRICALLY OPERATED ECCENTRIC DRIVE

Walking beam type electric furnace

Labels on figure:
HEATING ELEMENTS — AIR COOLING FAN — LEATHER BELTS TO PREVENT MARKING WHILST CHARGING — AIR RETURN DUCTS — AIR DISCHARGE NOZZLES — ROLLERS SUPPORTING ROPES — STAINLESS STEEL WIRE ROPES — COOLING AIR DISCHARGE DUCTS

Flash annealing electric furnace.

problem will be pleased to learn that apparently a satisfactory design has finally been produced and will be anxiously awaiting further details. Undoubtedly, this type of furnace has a definite inherent quality in its heat efficiency, and far surpasses the normal conveyor or pusher type. By virtue of its compactness and relatively small cross sectional area it has a lesser consumption than the rotary drum furnace. Non-productive weight is totally eliminated, effecting a considerable saving in power consumption. For the heat treatment of small components this type of furnace would appear to offer many advantages. Reviewing past developments, both on the Continent and later in this country, one finds that there have been many ingeniously devised methods of conveying charges over a flat tray, which can be summarised as follows :—

(1) Cam-operated drive with accentuated forward stroke.
(2) Cam-operated, with accentuated return stroke.
(3) High frequency vibrating motion by an interrupted electric supply.
(4) Crank oscillating movement fitted to the inclined supports of a level tray.

During the war, in December, 1941, METALLURGIA published an article by the present author on a new continuous-type electric furnace, claiming total elimination of non-productive work, simplicity of construction and method of handling, comparatively low initial cost and an unlimited field of application. This particular article dealt with many applications, and included reference to a method of recuperation, using the shaker hearth principle for an annealing plant. The level tray was supported at close intervals on vertical levers set at a slight angle, and connected to an electrically operated crank drive. The motion was, in fact, the principle of the parallel link, and the forward movement was obtained with the crank running at a constant speed. In operation the charge would appear to flow through the furnace in a consistent, even mass, and discharge with rhythmic precision. It was proved in later years, however, following exhaustive tests, that absolute consistency in hardness cannot be guaranteed, and whilst heat-treated work might be satisfactory in many cases, the results were not acceptable, as they did not satisfy the rigid hardness tests required by most industries. Later, it became very apparent that at the beginning and end of each batch of work, the rate of travel through the furnace could not be controlled, and consequently it was agreed that two like components were not subject to the same time-temperature cycle.

Difficulty of Ensuring Even Flow

Every conceivable type of tray, including corrugation, was considered and tried, but the problem was a complex one, and a successful solution was not obtained. This type of furnace, so simple in operation, undoubtedly possesses powers of performing unbelievable antics on the progress through the furnace of normal industrial charges of equal size and weight.

It is necessary to summarise the faults which come to light in practice, so that the writer may endeavour to prove the reason for the discrepancies, by means of reference to basic principles and accepted laws.

(1) At the commencement of each new batch of work, the first components would endeavour to race ahead to the soaking zone, gradually reducing speed until the main mass of work, which gradually increased in bulk, reached the " leaders," absorbing them in its travel.
(2) Similarly, at the end of each batch, the last components would slow up in the soaking zone, some actually sticking and necessitating forcible discharge.
(3) Occasionally work would jam in the tray, and over-riding of the components would take place. In numerous cases the obstruction would break up by the " shaker " movement, and the affected work pass through unobserved.

It is advisable, at this point, to explain that the tests were carried out in larger furnaces than now contemplated, and it is appreciated that smaller furnaces would have the tendency to minimise the difficulties explained.

Items 1 and 2 are related, and may be considered together. In the first instance it is necessary to consider the first law of friction : " Friction is proportional to pressure when the surfaces are the same." Depending on the method employed for charging, the depth of work on the tray may vary considerably, and the speed of travel will differ according to the frictional resistance. According to Goodman, frictional resistance on dry surfaces is not greatly affected by temperature, but when the surface is lubricated the resistance depends more upon the temperature than on any other condition.

From the foregoing, it is obvious that the problem is a complex one, and the difficulties present an almost insurmountable barrier. Most charges are not free from oil, and as the components pass through the furnace, lubrication becoming more meagre, the frictional resistance approaches the behaviour of dry surfaces. Their progress becomes sluggish, until the main mass of work proceeds more evenly, absorbing in its travel the " early starters." Comparing the physical nature of the two surfaces in contact, or the co-efficient of friction, which equals $\dfrac{P}{W}$ we have for example :—

Brass on C.I. (dry) = $0 \cdot 217$
Brass on C.I. (greasy) = $0 \cdot 017$

To the most unimaginative designer, it is obvious that these variations alone make calculations of speed impossible, and the rate of travel or time in the furnace depends purely on trial and error.

At a recent demonstration, the writer placed two nuts of equal dimensions on a shaker hearth furnace, having a smooth corrugated tray. It was interesting to note that, although both components were placed in direct line with one another, they were approximately a pitch and a half apart before they had entered the furnace, clearly demonstrating the unreliability and inconsistency of the method of conveying.

Deviating from the serious aspect for one moment, it will interest engineers to know that this method of travel has now been adopted to create a new and fascinating gambling game. The tray, which is perfectly flat and provided with a number of tracks, is vibrated by an electric battery. The charge comprises a number of horses or dogs of equal weight and dimensions, each having a smooth base. The variation of the progress during the race is very uncanny, and the result is always speculative.

Conclusion

Viewing, in true perspective, the design of heat treatment electric furnaces, the majority of the larger type involving mechanical handling, displays a crudeness so very strange to the experienced mechanical designer. It would appear that the finesse of mechanics is not yet fully employed, and it is expected that this period of " consolidation," already referred to, will enable superior designs to be produced with consequent saving in power, maintenance and costly labour.

Industrial Finishes Exhibition

In the past good design has largely been taken to mean satisfactory functional qualities in use combined with pleasing lines, but to-day there is a growing realisation of the importance of quality in material, workmanship and finish. Apart from improved appearance, the life of an article is often dependent on the finish and a bad finish means fewer repeat export orders. The remarkable advances in industrial finishing which have taken place in recent years are not perhaps, as widely appreciated as they should be ; the efforts of the organisers of the Industrial Finishes Exhibition should do much to remedy this state of affairs.

THE first National Exhibition of Industrial Finishes to be staged in this country will be opened by Sir Charles Goodeve, O.B.E., D.Sc., F.R.S., on August 30th, and from then until September 7th, visitors to Earls Court will have an opportunity of seeing the latest finishes and finishing plant. Considerable space will be devoted to the many and varied approaches of industrial finish, but the main emphasis will be on metal finishes. The Central Technical Exhibit will provide a complete survey of finishes available to-day, in all their applications, and takes the form of a series of displays, each covering one aspect of the finishing industry.

In former civilisations the range of colours available was limited by the available materials and the more brilliant effects had to be achieved by ingenuity of contrast, rather than by selection from a large range. The range of colours available to the manufacturer to-day is almost infinite, in any of the many forms of industrial finish, and visitors will find much of interest in the Hall of Colour, where there will be displayed a series of demonstrations covering a wide historical range of colours from Dynastic Egypt to the Sevres Pottery of the late 18th century. Actual manufactured objects will be shown together with an analysis of the colours used and their modern equivalent.

On the individual stands, the buyer or technician will be able to inspect the best equipment and processes at the command of industry and discuss paint, electroplating, vitreous enamelling, or whatever his problem may be, with experts in that particular field.

Stands of General Interest

The importance of finish to the export industries led the COUNCIL OF INDUSTRIAL DESIGN to support the venture from its inception. On the Council's stand (No. 34) examples of good and bad finishing of domestic equipment and machinery will demonstrate the wisdom of consulting the " finishing " specialist from the outset when planning a new product. Several new and interesting finishes will be displayed, including a new
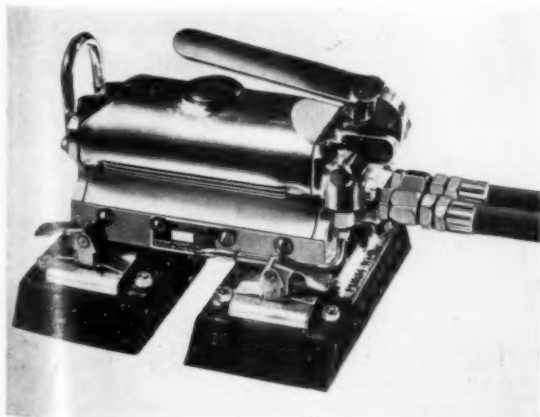
Sundstrand Model 1000A Wet Sander
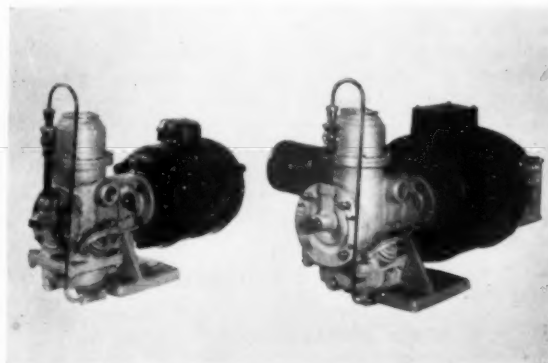
Keith-Blackman type OA (left) and IA Compressors
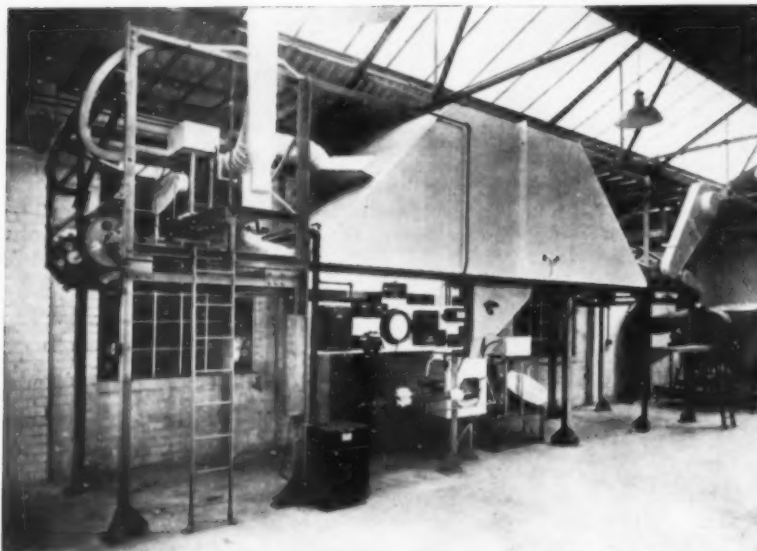
Conveyor enamelling plant.
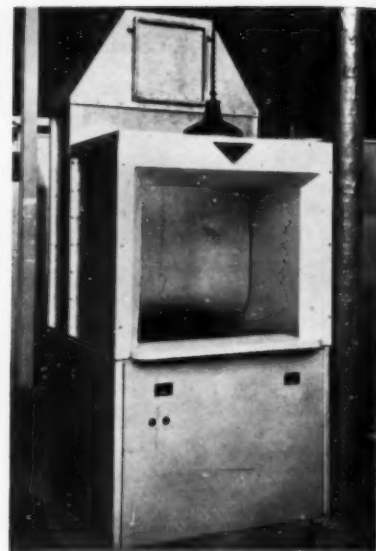
Water wash spray booth.

high-temperature resisting enamel finish ; a high quality coloured electro-brightening finish on die-castings ; an enamel plating shown in a variety of colours ; a vitreous enamel, free from lead which can be applied to aluminium ; white and cream coloured anodising and speculum plating.

In order that some measure of the quality of finishes may be established, the BRITISH STANDARDS INSTITUTION has drawn up a number of specifications for industrial finishes. The complete range of such British Standards will be on view on the Institution's stand (No. 66) covering such varying items as : Deposition of metallic coating on metallic articles by various means ; Methods of tests for vitreous enamel finishes ; Colour standards for paints, distempers and vitreous enamels. In addition, apparatus and equipment used in the testing of industrial finishes will be featured. Most of this equipment is already referred to in the appropriate British Standard, but a few items are under consideration with a view to inclusion in future standards.

On the commercial side, the BOARD OF TRADE are organising an information stand (No. 55), in conjunction with the Technical Information Documents Unit, to deal with enquiries on traders' problems, and particularly with difficulties arising in export trade.

Members and friends of the ELECTRODEPOSITORS TECHNICAL SOCIETY will find a meeting place on Stand No. 10 where the Society will show a complete set of the Volumes of its Journal, along with charts illustrating the growth of the Society and the range of subjects dealt with at its meetings.

In addition, there will be a number of non-technical service stands including a Theatre Ticket Bureau on Stand No. 73 (KEITH PROWSE AND CO. LTD.).

Plating and Polishing

The display of electroplated parts to be shown by ATLAS PLATING WORKS LTD., on Stand No. 29, is designed to show mainly the results of new methods of plating and preparation resulting in a saving on production costs. Of special interest in this connection will be a display of parts burnished by the Roto–Finish barrel-finishing process* which eliminates hand polishing, and a display of small parts chromium plated by a barrel method. Other exhibits serve to indicate the finish to be obtained on the centreless polishing machine ; the successful results obtained in chromium plating zinc-base die castings ; the use of hard-chrome for gauges, press tools and worn parts ; and a new satin-chrome finish developed for the photographic trade.

A wide range of decorative and protective finishes will be shown on Stand No. 9 by the HOLMES PLATING Co. LTD. Emphasis will be laid on the adaptability of modern plating methods in relation to mass produced articles which results in high quality speed of production and low costs. Particular attention has been paid to the importance of adhering to the B.S.I. and Metal Finishing Association specifications. The display will include chromium, nickel, gold, silver and rhodium on steel, brass, copper and zinc-base alloy in polished, dull or satin finish.

The only firm of industrial metal polishers at the exhibition will be the GOLDNEY METAL POLISHING Co. whose exhibits will include a miniature set of tubulas furniture showing metal finishes ; aluminium castings before and after polishing ; and a compressor sprayer with spray gun, of the type used for shop fronts.

An interesting range of up-to-the-minute electro-plating and metal finishing processes will be exhibited by M. L. ALKAN (SALES) LTD., along with a new range of electrical control apparatus for electroplating. The latter will include new methods of remote resistance control ; automatic time control of plating and cleaning cycles, and time controlled current reverse units of capacities up to 1,500 amperes. Several American finishes of which the Company holds sole rights will be shown including Promat bright zinc, Enthone Ebonol " C " blackening for brass and copper alloys and Tygon

* For a description of the process see *Metallurgia*, November, 1949, 33–34.
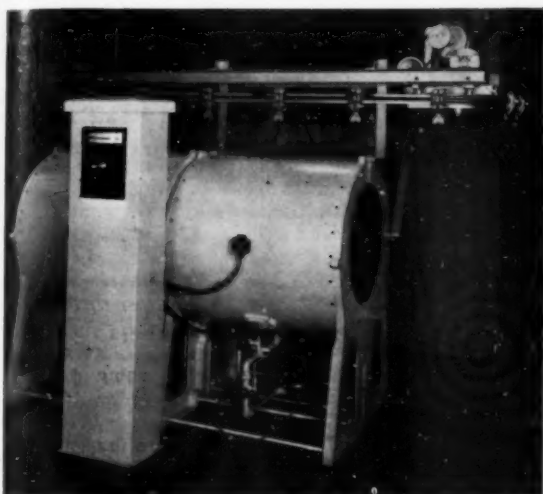
Courtesy of Parkinson & Cowan (Gas Meters), Ltd., and Teleflex Products, Ltd.

Tunnel-type infra-red dryer with Teleflex overhead monorail conveyor

coating materials for plating racks. A full range of Alkan anodes and a comprehensive display of polishing equipment will also be on view.

The main theme of the display on Stand 36 (GRANER AND WEIL LTD.) centres round a comprehensive range of electroplating and polishing equipment. The exhibits will include scratch brush and polishing lathes ; polishing mops which will include the latest developments in stitched, pleated and bias flow types ; polishing compositions ; fibre and wire wheels ; and several grades of hand scouring brushes. The plating plant on show will comprise G. and W. Westalite metal rectifiers ; control panels ; a complete nickel plating vat ; a horizontal rumbling barrel ; two new plating barrels, both of variable angle ; motorised pressure filters ; and a range of plating salts and anodes.

A main feature of Stand No. 32 will be drawings and designs while you wait. JOHN PRESTON AND CO. LTD. specialise in the design and manufacture of plating jigs of which they have produced more than 2,000 types. There will be jigs designed for copper, nickel, chrome, silver, bright nickel and chrome, anodising, etc., and the various classes of jig will include all-purpose jigs, spring-frame jigs, magnetic jigs, replaceable-bar magnetic jigs, and replaceable hook jigs.

The main purpose of the exhibits of SILVERCROWN LTD. on Stand No. 22, is to introduce the latest and most efficient methods in the plating and polishing fields. A semi-automatic plant for both acid and alkaline solutions will be on view. This plant can be heated by gas, steam or electricity, with timing arrangements for 20, 30 or 40 minutes. Other plating plant will comprise : continuously operating small size plating barrels ; a totally immersed plating barrel ; and resistance boards and rectifiers. For pre- and post-plating operations there will be a rumbling barrel ; polishing mops and compositions ; and a back stand idler working with finisher bands. Plating chemicals and cleaners will complete the display.

Finishing Plant

Although most of the plant shown at the Exhibition is, strictly speaking, finishing plant, that dealt with in

Courtesy of Atlas Plating Works, Ltd.

Roto-finished articles.

this section will be concerned with the application or after treatment of paints, enamels (other than vitreous), lacquers, etc.

The AERASPRAY MANUFACTURING CO. LTD. has a long established connection with spray painting and on Stand No. 58 the visitor will not be surprised, therefore, to find a full range of spray guns, and rectifiers, pressure feed containers, and compressor sets. A recent development in spray painting is the Ransburg Electrostatic process, in which the paint is attracted to the article by electrostatic means, thus avoiding waste. Automatic spray guns and fluid pressure regulators for this process will also be on view. Of special interest will be a new type of water-wash spray booth with several notable features, including water curtains at the sides as well as the back, and a highly efficient washing chamber using unchokable nozzles in combination with internal volutes giving maximum separation of paint particles from the air stream.

The belief that " the proof of the pudding is in the eating " lies behind the design of Stand No. 45, where F. J. BALLARD AND CO. LTD. will be showing a working unit conveyor enamelling plant. The equipment is as supplied for production purposes and the object of the exhibit is to place at the disposal of visitors, clients and their friends, a unit which is available for testing on site, under actual working conditions, any samples that may be submitted.

Fans form the basis of several exhibits on KEITH BLACKMAN's Stand, No. 5. A special type with laminated plastic blades is suitable for the removal of fumes of a corrosive nature, whilst the " Weldafume " unit is specially designed for removing hot fumes and smoke from the vicinity of welding operators. A self-contained
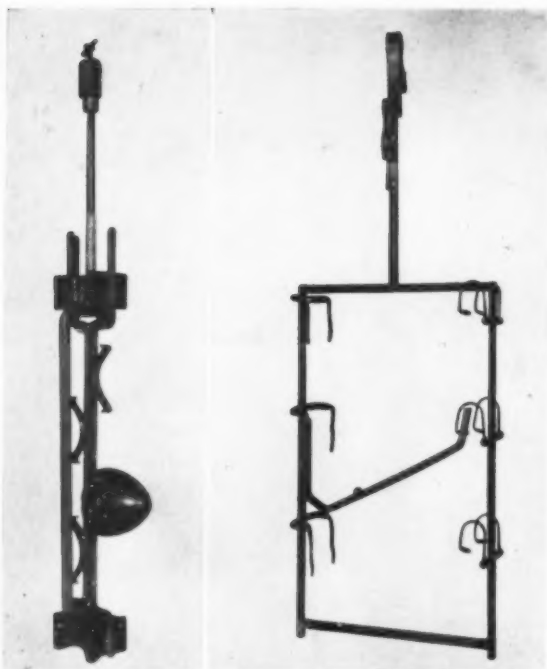
Courtesy of John Preston & Co., Ltd.

Left : Lamp body magnetic replaceable bar jig for bright nickel and chromium.

Right : Window channel jig for bright nickel and chromium.

apparatus for dust exhaustion and collection from general grinding and light polishing machines will find application in metal finishing shops. Other exhibits will include compressors for handling air or gas, for pressure or suction, up to 5 lb./sq. in. ; burners ; thermostats ; and a combustion chamber for high temperature drying by products of combustion and excess air, using town's gas as fuel.

The basis of LIONEL HOOK & SONS' show, on Stand No. 57, will be their " Cotswold " dipping machines, many of which have been supplied for colouring metal ware. The articles to be dipped are suspended on an adjustable overhead platform, the container of fluid being lifted to the work by means of a ram, operated hydraulically by a simple and efficient force pump. The standard capacity is 20 in. but this can be increased to 24 in. if required. A larger model, operated by a power driven pump unit will also be on view.

Infra-red heating units have proved very suitable for the storing of paint on all types of metal components ranging from compact cases to large compressor engines. On Stand No. 65A PARKINSON & COWAN (GAS METERS) LTD. will be showing two types—tunnel and panel—of heater for this purpose. Each is made in units 3 ft. long, the tunnel type being available in three sizes, 2, 3 and 4 ft. diameter whilst the panel type may be 36 in. wide by 18 in. high, or 27 in. wide by 13½ in. high. The burners are of the non-aerated Bray jet type, secondary air for combustion being introduced through air inlet holes. The Teleflex overhead monorail conveyor will be shown in conjunction with these units.

Feature of the THOMAS DE LA RUE exhibit, on Stand No. 65B, will be an 18 panel octagonal infra-red drying

tunnel suitable for storing either black bituminous paint or the most delicate pastel shades. Temperatures up to 750° F. may be attained in the tunnel shown, thus providing a valuable margin when really heavy articles are to be dried. Maintenance is negligible and experience has shown that the actual gas consumption is 1·3 therm/hr. under working conditions. The universal application of the Potterton panel is best demonstrated by the fact that it is used for both the baking of the enamel on fine armature wire and the drying of paint on 13-ton railway coal wagons.

For the high grade finishing of paint, cellulose, wood, metal and plastics, SUNDSTRAND PNEUMATIC SANDERS (BRITAIN) LTD. will be demonstrating a portable pneumatic sander on Stand No. 70. Flat or curved surfaces may be tackled easily by using one of the several types of pad attachment. Patented rubber pads automatically mould themselves to curved surfaces, whilst hard flat pads are used where a flat surface free of ripples is desired. The Model 1000A is fitted with a water feed for wet sanding.

Chemical and Electrochemical Finishes

The exhibit of ALTONES LTD. (Stand No. 69) is possibly unique in that the whole stand is made from the material it is designed to show. Altones Ltd. specialise in the fabrication and surface treatment of aluminium and its alloys. On the stand may be seen aluminium in all its forms—sheets, bars, tubes and special sections—in a variety of beautiful colours and in many different surface treatments. One side of the stand takes the form of a cocktail bar whose decorative finish is proof against spills and cigarette burns, whilst another will feature customers' material after processing Yet a third display will include products designed, manufactured and finished by the Company.

There is a growing realisation of the import contribution which can be made to the improvement of paint coatings by effective pre-treatment of the metal. On Stand No. 51, JENOLITE LTD. will be demonstrating their Jenolising Process, whereby rust removing, rust proofing, phosphating and paint binding are effected in one operation. Also shown will be the Jenolising Process for aluminium by means of " Jenolite " Aluminium Keying Solution which removes the oxide skin and provides a key for subsequent painting and enamelling. A new product shown for the first time will be " Jenolite " Aluminium Degrease and Cleaner, A.C.1, which will clean and degrease aluminium without any attack on the metal.

Component parts treated by a wide range of chemical immersion processes will be exhibited by METAL PROCESSES LTD., on Stand No. 60. The Mark II Black finish for steel is an effective rust resister and paint base. Other coloured finishes for iron and steel include Oxydalin, W.I.S., Ruskil, Gunsmith's Black and Goldit. On certain copper alloys an imitation nickel plate can be obtained by immersion in Niklit. Other finishes shown will include blacks, gold, bronzes, blues and steel greys on copper alloys ; blacks and other colours on zinc and Mazak products ; and black, grey, yellow, bronze, brown, mottled and iridescent colours on aluminium alloys. For cleaning a non-caustic cleaner, Degres, will be shown, whilst for transit of brightly finished metal articles, Meprol rust preventive dipping solution will be featured.

Reference has already been made to the exhibits of

M. L. ALKAN (SALES) LTD., which include a number of chemical finishes.

Organic Finishes

A few years ago, LEWIS BERGER (GREAT BRITAIN), LTD., introduced a new range of decorative finishes called Bergermaster, based on Styrene, which proved very successful. They are now introducing a new range of industrial finishes, all based on styrene, which will be known as Polykem, and will cover, eventually, all the various fields of industrial finishing. Being based on styrenated alkyds, they have several advantages over normal finishes, including quicker drying.

The FERGUSON EDWARDS stand, No. 23, will display, in applied form, the various " Edlac " industrial finishes. The articles shown will be selected at random from clients' factories with no further special finishing. Ferguson Edwards, Ltd. believe that users should adopt the use of testing equipment to obtain maximum control and consistency in the production line ; with that end in view, demonstrations of various items of testing equipment will be given. As the firm have an Industrial Colour Advisory Department, they will display " Edlac Cromotonics " showing the scientific application of colour to machinery and factories.

The production of cellulose acetate products has been a speciality of HAREFIELD INDUSTRIAL PAINTS LTD. for some time and amongst the exhibits will be insulating varnishes and cable lacquers. Included in the industrial finishes visitors will find types suitable for car and bus bodies, refrigerators and metal cabinets, together with chromium protective lacquers. The spot welding primer shown can be applied to sheet metal prior to spot welding without appreciable loss in strength of the joints. It is formulated for spray or dip application and is supplied for air drying or stoving. It is sometimes desired to leave parts of a surface uncoated in stove enamelling ; this can be effected by pre-treatment with a Stripper Base which deteriorates on stoving and allows the coated section to be brushed clean.

The variety and versatility of JENSON AND NICHOLSON LTD.'s industrial finishes will be demonstrated on Stand No. 44B by means of beautifully finished models which show, in a nutshell, the main industries served and the types of enamels, lacquers, paints and varnishes most suited for particular purposes. Among the finishes shown will be Lustralac polychromatic finishes ; black and coloured stoving enamels ; Solventless, Coilac and Lacwatt insulating and impregnating varnishes ; Mafinel machine enamels ; Seaborne marine finishes ; Jenorotin tinplate finishes ; and the revolutionary 30-minute Rivelling enamel. Examples of the time, labour and cost saving properties of the new J. and N. wash primer will be shown. This is a lightweight primer which bonds perfectly to any metal and inhibits corrosion. One gallon covers 100 sq. yds. and products treated with it are ready for stoving enamel or cellulose lacquer in 15 minutes.
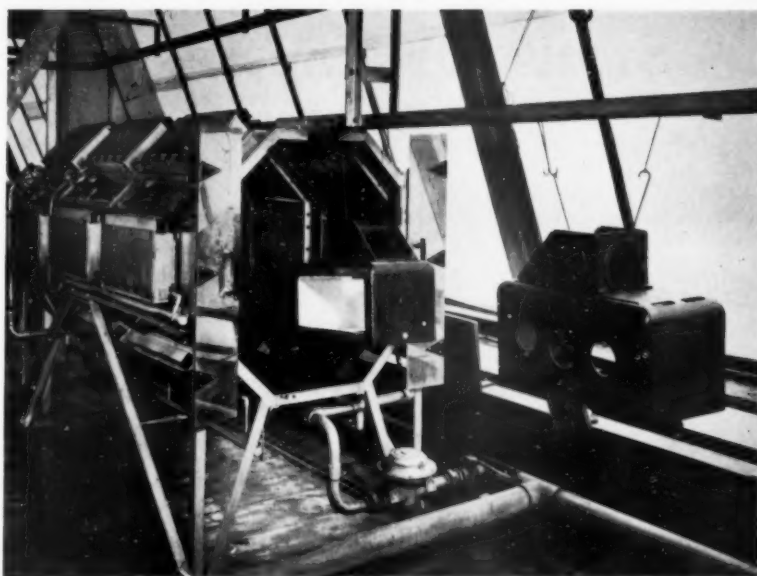
Courtesy of Charles Baker, Ltd., and Thomas de la Rue & Co., Ltd.

Infra-red drying of wrinkle finishes on epidiascope castings and scientific instruments.

PHENOGLAZE LTD., a pioneer in marketing cold setting plastic compounds for the last fifteen years, will be exhibiting examples of this type of finish on Stand No. 71. They are manufactured under the trade names Pheenoglaze, Pheenorock, Pheenalite, Pheenopol, Pheenoflex and Pheenoment. Each has its special properties to allow for the different requirements of industry. By varying the catalysts used for hardening polymerisation it is possible to vary the finish from a very hard one to a flexible one. Recent developments include A.E.R.E.61 and 62 which are particularly suitable as protective coatings against radioactive dust which can easily be removed from the very dense finish.

The important role now being played by the chemicals-from-petroleum industry in the production of surface finishes will be demonstrated on Stand No. 67 by SHELL CHEMICALS LTD., who are pioneers in this field. The stand has been largely decorated with finishes in the manufacture of which Shell chemical products have been employed and there will be a number of panels coated with lacquers made with Shell ketones. In pictures and charts related to the Shell chemical plant at Stanlow, due emphasis is given to products of particular interest to the surface coating industry, such as acetone, methyl ethyl ketone, methyl isobutyl ketone, diacetone alcohol and secondary butyl alcohol. The well known Shell wetting agent " Teepol " will also be shown to have its place in finishing by virtue of its use in pigment grinding, stabilising distempers and degreasing prior to coating, plating and acid pickling.

The TEMPLE VARNISH CO. LTD. stand, No. 52, will reflect the increasing importance of the liquid coating industry. Limited space precludes the showing of the full range of products, but the examples chosen, which include a refrigerator, washing machine, and motor body and general industrial and decorative finishes, will serve to illustrate the general range. The preparation of these finishes, the result of extensive research, involves the

Steels for Tapered Roller Bearings

Factors Affecting Manufacture and Service

By J. H. Evans, F.I.M.

Chief Metallurgist

A four-day Symposium on Tapered Roller Bearings was organised recently by British Timken Ltd., and attended by members of the Engineering Staffs of various Universities and Technical Colleges. During the course of the Symposium, opportunity was afforded of seeing the manufacturing and inspection processes involved in the production of ball and roller bearings, whilst a number of papers concerning design and manufacturing problems were presented by members of the company's technical staff. The question of materials was dealt with by Mr. Evans, in a paper on " Some Metallurgical Aspects of Steels for Tapered Roller Bearings," a slightly abridged version of which is presented here.

THE efficiency of the modern tapered roller bearing is due in no small measure to the high quality materials used in its manufacture. The maintenance of this high quality necessitates an elaborate metallurgical inspection procedure which is, however, fully justified when reliability and performance in the field are factors of primary importance.

Since their inception, tapered roller bearings have always been made from case-hardened steels, the combination of a hard wear resistant case and a tough shock-resisting core being eminently suitable for withstanding the heavy localised and shock loads to which the bearings are so often subjected in service.

General Considerations

Before considering the more detailed metallurgical aspects, certain factors affecting the life and performance of tapered roller bearings may be enumerated.

1. In view of the severe service conditions under which they have to operate, it is essential that the hardened surfaces are free from soft spots and of the greatest degree of hardness conducive to maximum wear resistance under the most exacting conditions, whilst dimensional accuracy must be maintained throughout the temperature range for which the bearings are designed to operate.

2. The steel must be free from harmful stringer-type inclusions, a requirement in itself necessitating stringent examination of all incoming materials.

3. Consecutive deliveries of steel must respond to heat treatment in a uniform manner, and exhibit the best mechanical properties obtainable with the particular analysis used, whilst distortion in machining, or during heat treatment, must be at a minimum to enable close dimensional tolerances to be maintained throughout production.

4. The bearings must be economical to produce, a feature which is dependent on the cost of the material, the machining properties of the alloys used and their behaviour during heat treatment. It will be appreciated that these high quality alloy steels are expensive, and it is, therefore, of the utmost importance that the highest degree of machinability is attained with the least distortion possible, so that some economy may be effected in subsequent operations—thus resulting in greater productivity—in order to offset, to some extent, the initial high cost of the raw material.

Metallurgical Requirements

Cleanliness.—The commercial production of steel without inclusions being an ideal still to be achieved, a standard of cleanliness has to be adopted which will result in satisfactory service of the bearings made. Several slag counts or charts have been introduced, from time to time, which give a numerical valuation for each type and thickness of inclusion. One of the most suitable is that compiled by the Swedish Jernkontoret, which has been adopted as a tentative standard by the American Society for Testing Materials. Line 1 on this chart, which represents the highest degree of cleanliness obtainable on a commercial basis, may be regarded as the general acceptance standard for steels for tapered roller bearings. Under no circumstances are isolated stringer-type inclusions worse than those represented by 2A or 2C accepted.

The method of inspection usually employed starts with an examination of sections, cut from each bar or tube, after deep etching in boiling 50% hydrochloric acid. Sections showing the etching pits indicative of inclusions are sectioned further and examined microscopically. The long stringer-type inclusions are considered to be the most dangerous, as when they break the surface they are likely to act as nuclei from which disintegration may start in service. There is, however, little direct evidence to prove the actual effect of the different types of inclusion on bearing life and performance, but as a safeguard to ensure that only the highest quality material is used, all types of inclusions are treated as equally dangerous.

Analysis.—Consistent machinability, close dimensional control, and satisfactory hardness and mechanical properties can only be assured if all the alloying elements which may be present are held to much closer limits than normal uses warrant.

Machinability.—This is an evasive property, not often subject to control by any one factor, and no truly conclusive machine test has yet been devised which gives more than an indication of some of the problems involved. It is a function of analysis, melting practice, grain size, method of processing, mechanical characteristics, relative hardness and metallographic structure.

For present purposes, machinability may be considered as pertaining to the actual machining operations on like machines, under similar conditions of feed, speed and coolant, but as affecting the dimensional accuracy

obtained among like pieces and the number of pieces per tool grind in actual production, co-related to the relative hardness, grain size, analysis, and metallographic structure resulting from different normalising or annealing operations.

Experience indicates that there is an optimum hardness and brittleness produced by the alloying elements present in the steel which give the best machining properties for any particular chemical analysis. Expressed in terms of Brinell hardness, the range 183–217 is generally found to give the best results.

Because of the influence it exerts on the structure and mechanical properties, the chemical analysis is one of the major factors controlling machinability. Elements usually considered as carbide formers behave in a similar manner to carbon but, because of the hardening effect, the strength of the material becomes too high for good machining. Fortunately, this can usually be offset by suitable modification of the softening treatment. Elements forming solid solutions in the ferrite tend to have a deleterious effect on the machining properties, on account of the strengthening and toughening influence they have on the ferrite. This cannot be completely eliminated, particularly with the higher nickel alloys, but considerable improvement can usually be brought about by controlling the rate of cooling after hot rolling or forging.

Grain Size.—The case-hardening steels now used are of the inherently fine-grained type. This results in the following advantages : Heating temperatures are not so critical; dangers of cracking or warping during hardening are minimised; a greater toughness is obtained for a given strength and hardness; less grain growth occurs during the carburising operation, so that parts may be quenched direct from the carburiser without subsequent heating to refine the core.

Being tougher than corresponding steels with a coarse structural grain, they are a little more difficult to machine, but this deficiency can, in most instances, be overcome by suitable heat treatment prior to machining, while the slightly shallower depth of hardening can be corrected by the addition of suitable alloying elements.

Hardenability.—By a careful balance of certain of the alloying elements, the hardenability of the steels used, as measured by the Jominy End-quench Test, is carefully controlled.

Factors Affecting the Choice of Case-Hardening Steels

It will be appreciated from the foregoing that considerable care and attention must be given to the selection of suitable case-hardening steels for the production of tapered roller bearings. The cleanliness requirements necessitate the use of high quality stock irrespective of the alloying elements present. Of the other factors, those influencing the strength and resilience of the core are most important, as they determine the type and amount of alloying elements which may be needed.

The core must have sufficient strength, after suitable heat treatment, to withstand the heavy crushing loads imposed in service and also the compressive stresses induced in the case during hardening, whilst it must be resilient enough to accommodate shock loads without permanent deformation. Generally speaking, the magnitude of the stresses to which the bearing will be subjected dictates the mechanical properties of the core as well as the metallographic structure and depth of the case.

The choice of analysis is also dependent on the availability of the alloying elements selected, as the steel must be readily available from alternative sources, whilst its machining properties and response to heat treatment must be consistent from cast to cast, to ensure uniform hardenability of case and core, together with close dimensional control throughout continuous production.

Of the alloying elements which may be introduced some, such as chromium, molybdenum and manganese, tend to form carbides and, therefore, have a direct influence upon the strength of the steel and its hardening power, whilst others, of which nickel is a typical example, go into solid solution in the ferrite and thereby impart toughness, strength and improved shock resistance. Combinations of both types of elements possess the individual characteristics of each and, after carburising and hardening, have a high elastic limit combined with good ductility and hardness, which add up to high strength and resilience in the core and maximum hardness characteristics in the case. From the very wide choice of alloy steels available, brief mention will be made of two which have been proved by experience over the years to be the most suitable, and which are now universally used for the production of tapered

TABLE I.—MECHANICAL PROPERTIES OF BEARING STEELS

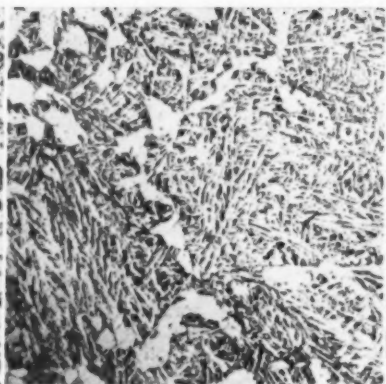| Material	Treatment	Mechanical Properties					
		Yield Tons Per sq. in.	Max Stress Tons per sq. in.	Elongation %	Izod Ft./lbs.	Reduction of Area %	Brinell Hardness
Nickel-Molybdenum Case Hardening Steel	Oil Quenched 900° C. Oil Quenched 795° C. Stress Relieved 165° C.	40·5	62·1	22	58–62	49·0	285
	Air Cooled 900° C. Oil Quenched 860° C. Oil Quenched 795° C. Stress Relieved 165° C.	40·6	62·4	22	60–63	49·6	277
" Krupp " Nickel-Chromium Case Hardening Steel	Oil Quenched 900° C. Tempered 6 hours 595° C. Oil Quenched 790° C. Stress Relieved 165° C.	70·9	80·1	17·5	70–76	60·4	363
	Air Cooled 900° C. Oil Quenched 840° C. Tempered 6 hours 595° C. Oil Quenched 790° C. Stress Relieved 165° C.	70·6	80·6	18·3	69–74	60·2	363
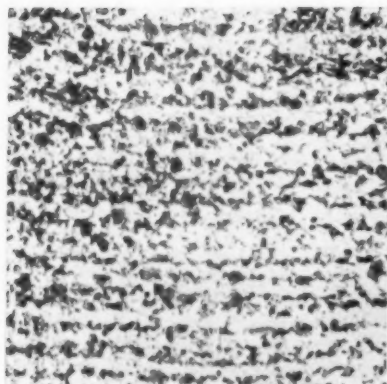
Fig. 1.—Nickel-molybdenum steel, hot rolled. Brinell 187. × 100.
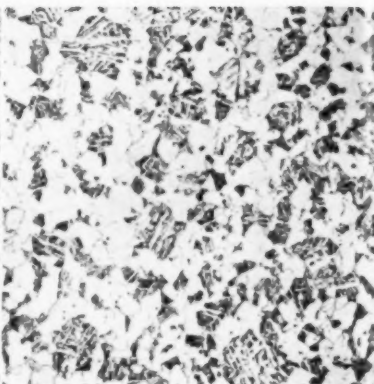Fig. 2.—Nickel-Molybdenum steel, hot rolled. Brinell 229. × 100.
Fig. 3.—Nickel-molybdenum steel, "Blocky" structure. × 100.

roller bearings. They are the nickel–molybdenum and nickel–chromium case-hardening steels.

Nickel-Molybdenum Case-Hardening Steel

Specially developed by British Timken's American associates for all automotive and general purpose bearings, this steel does not conform to any standard specification, although it may be considered as a modification of the American S.A.E. 4620, whose British equivalent is En. 34.

It is a fine grained steel, with an A.S.T.M. grain size of 5–8 made to controlled hardenability, which is achieved by close chemical limits for all alloying elements, particularly the carbide formers. A typical analysis is indicated below :—

C	Si	S	P	Mn	Ni	Cr	Mo
0·20	0·18	0·019	0·018	0·55	1·73	0·28	0·21

An indication of the mechanical properties which may be expected in the finally hardened core is given by the figures in Table I which records the result of tests on 1 in. diameter bars treated as shown. The first series of treatments corresponds to that given to gas carburised parts which are quenched from the carburising heat, reheated to refine the case, and stress relieved.

Pack carburised parts are cooled to room temperature in the containers before the quenching treatments as shown by the second series of treatments. That the high temperature quench from 900° C. does not adversely affect the mechanical properties will be seen from a comparison of the test results, which are almost identical.

Nickel-Chromium Case-Hardening Steel

This is familiarly known as "Krupp" steel. It is pre-eminent among case-hardening steels, and when fully hardened is capable of withstanding the most severe loading conditions ; it is therefore, used for all heavy duty applications.

Like the nickel molybdenum steel it was specially developed by British Timken's American associates for specific purposes, and there is no exact equivalent among the standard specifications. It is somewhat similar to S.A.E. 3312 and En. 39A, but the main alloying elements are held to much closer limits, whilst it is also supplied to a specified grain size, which enables an accurate control of the hardening to be maintained.

A typical analysis is indicated below :—

C	Si	Mn	S	P	Ni	Cr	Mo
0·12	0·20	0·38	0·018	0·016	4·01	1·4	0·08

In the heat-treated condition it possesses exceptional properties, exhibiting a high elastic limit and maximum stress, together with good ductility and the capacity of resisting fracture under shock loads, attributes which render it eminently suitable for the purpose for which it was developed. Typical mechanical properties are given in Table I. The treatments correspond with those for gas and pack carburising, again with almost identical results.

Metallographic Structure and its Effect on Production

Probably the most important metallurgical factor affecting production is the metallographic structure of the raw material, and as the treatment required to produce the best structure for machining differs for the two steels, they will be considered separately.

Nickel–Molybdenum Steel.—The hardness and micro-structure after hot rolling are dependent on the finishing temperature and the rate of cooling. In practice, Brinell hardnesses between 187 and 229 have been found in the same delivery, with microstructures similar

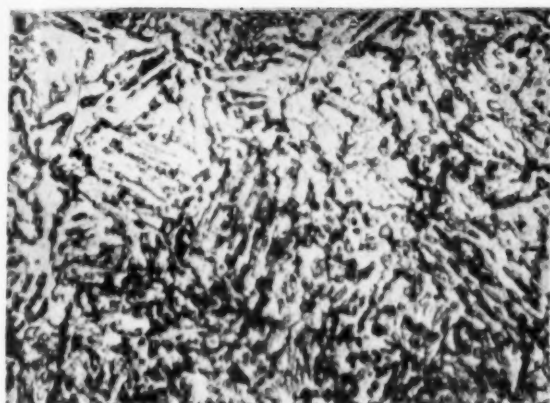Fig. 4.—Nickel-molybdenum steel, "Blocky" structure. × 1000.

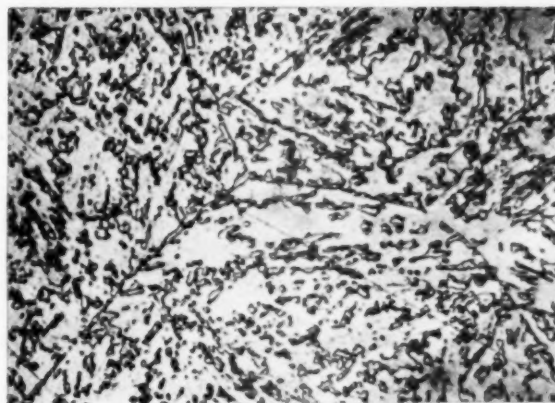Fig. 5.—" Krupp " steel, annealed, as received. Brinell 197.　　× 1,000

Fig. 6.—" Krupp " steel, slowly cooled from 980° C. Brinell 285.　　× 1,000

to those seen in Figs. 1 and 2. Such wide variations are obviously undesirable and not conducive to maximum production or close dimensional tolerances.

Considerable attention has been given to the production of the best structure for machining, and it has been established that that resulting from controlled cooling after hot rolling, particularly through the critical range, gives the best degree of machinability. Cooling from a high rolling temperature coarsens the grains, and the slower rate through the critical range develops a metallographic structure which has been termed " blocky," and which has a Brinell hardness range of 146–187. This structure, shown in Figs. 3 ($\times$ 100) and 4 ($\times$ 1,000), may be described as resulting from " partial spheroidisation of the carbides within the pearlite grain boundaries," in contrast to the uniformly dispersed spheroidal carbides in the case of complete spheroidisation resulting from prolonged sub-critical annealing.

The " blocky " structure enables accurate dimensional control to be maintained during machining, and also after final hardening. Advantage is taken of this to reduce grinding stock, with a corresponding increase in productivity in the Grinding Department.

All tubing is cold sized before use and, although this may increase the Brinell hardness to approximately 230,

no detrimental effects on the machinability have been observed.

Nickel–Chromium Steel.—The softening of the more highly alloyed nickel–chromium steel presents a different problem. Due to its air-hardening properties, a special duplex form of annealing is required to reduce the hardness to within machinable limits. This steel has always presented an appreciable amount of trouble and difficulty in machining, irrespective of the hardness, and experience suggested that the machinability was more influenced by structure than by hardness.

As delivered by suppliers, in the fully annealed condition, the structure has often been found to be similar to that shown in Fig. 5, from which it will be observed that although the carbides are partly spheroidised, there is still evidence of an acicular formation, apparently persisting from the decomposition of martensite. The Brinell hardness of 197 represents good commercial annealing and is well within the specification limits.

It has been found by experience that a fully spheroidised structure is ideal for machining this particular alloy, and although a Brinell hardness of 197 is usually associated with such a condition, this is not always the case. The usual annealing cycle is to heat to 980° C. and after soaking for a predetermined period—dependent on
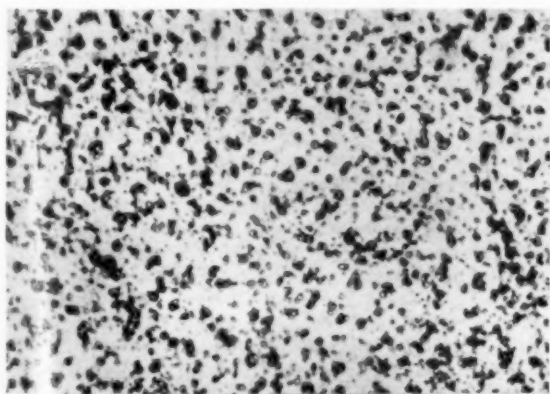

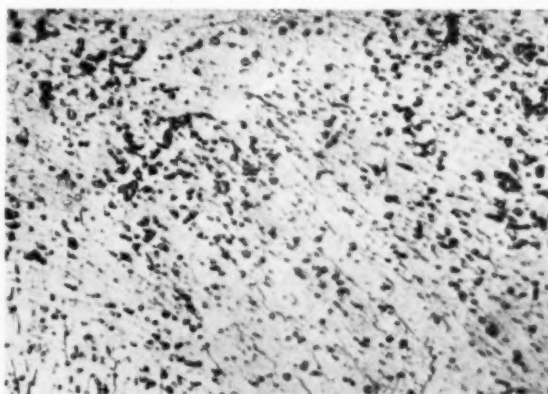Fig. 7.—" Krupp " steel, after first 12 hours temper at 640° C. Brinell 192.　　× 1,000

Fig. 8.—" Krupp " steel, after second 12 hours temper at 640° C. Brinell 187.　　× 1,000

the maximum section of the part—slowly cooling to about 100° C. before tempering to the desired hardness. To obtain a spheroidal structure, the tempering temperature should not exceed 650° C.

Part of the bar whose structure is shown in Fig. 5 was re-annealed to produce a correctly spheroidised structure. After heating to 980° C., soaking and slowly cooling, the bar was tempered at 640°C. for 12 hours. The structure after this treatment was almost completely spheroidised and a further 12-hour treatment completed the spheroidisation. The structures obtained after each stage are shown in Figs. 6, 7 and 8.

Carburising and Hardening

Neither steel presents any great difficulty in carburising and hardening, particularly as most of the furnaces used are electrically heated and capable of accurate temperature control. Both steels are either gas or pack carburised and, being fine grained, can be oil quenched directly, although with pack carburising the usual practice is to cool the parts to room temperature in the containers. When hardened and tempered to relieve residual stresses, the surfaces have a Rockwell hardness of 58–65 C scale.

Most of the small parts are quenched in machines specially designed for the purpose, whilst quenching jigs and fixtures are used for the larger parts ; in both cases the underlying principle is to control contraction the main object of which is to minimise distortion.

One of the most important advantages of the nickel–molybdenum steel is the close dimensional control which can be established by time quenching, that is, by allowing the component to remain on the jig in the machine for a predetermined time during the actual quenching operation. This method of quenching is being developed and will eventually be applied to most of the parts made from this steel.

All Krupp steel parts are tempered between core and case refining to break down any residual austenite in the case and to relieve primary quenching stresses.

The case depths employed vary between 0·040 in. for the smallest component to about 0·240 in. for the largest rolling mill bearings. The carburising times required to produce these case depths are approximately 10–168 hours for pack carburising and approximately 6–120 hours for gas carburising. The case depths are measured on actual components for the smaller sizes and true sections machined from material from the same cast for all Krupp steel parts.

Acknowledgments

Correspondence

THAT SPECIFICATION : MASTER OR SERVANT

The Editor, METALLURGIA.

Sir,

May I, through your columns, make some reply to the clear cut and refreshing article enunciated by Dr. Crow in your June issue ? In his article he mentioned on two occasions the use of non-ferrous work-hardened strip by manufacturers of electrical devices, and in both of his suppositional examples, the responsible parties, i.e., inspectors and draughtsmen, are held by him to be both unimaginative and incapable of thinking along lines other than those separating one specification from another.

Presumably both of these parties have contact with laboratories, who probably recommend the draughtsmen on their choice and the inspectors a decision on a questionable batch. In my own experience, the attitude of laboratories is to recommend usage of sub-standard material when, as a result of experience and practical try-outs, this is at all possible. What Dr. Crow has failed to mention is the case when material is supplied with borderline specification properties between two grades. In one case a certain quantitative property, hardness, was found to vary in the various strips from the mid-point of one temper to the mid-point of the next. When these figures were offered as the basis for rejection, the supplier pointed out that the average of the results was in the lower temper and thus the consignment was usable ! Obviously, a practical tryout is the first thought, but a quantitative reason is demanded when rejection is found to be necessary with material unsuitable in practice.

No, Sir. I do feel while Dr. Crow has presented an admirable essay on the use and position of specifications, the thought that users are hidebound ignorants, carrying copies of a B.S.S. in one hand and a rejection tally in the other is as erroneous as the thought that suppliers are incapable of producing the material ordered.

Yours faithfully,

Uxbridge Road,   C. G. WILLIAMS.
Slough, Bucks.
July 6th, 1950.

Comments on the concluding part of Dr. Crow's article from Mr. Williams are given in a further letter which is reproduced below.—EDITOR.

The Editor,

Sir,

I have read the concluding article by Dr. Crow and would not join issue with him in his suggestions for modifications of the relevant B.S. specifications, except to point out to Dr. Crow that no wording or phrasing of any specification will help to prevent a " pound-of-flesh " man from obtaining his 14 ozs. We accept them as an evil by-product of the age and are treated accordingly.

Dr. Crow mentions the advisability of practical tests. From a consumer angle, surely no material is rejected if it passes a practical test when its quantitative properties are sub British Standard specifications ? No sir, I cannot help but feel that Dr. Crow is thinking along the lines he has set out to destroy. No sensible person does think in the " Specification Habit," but it must be realised that, although capable of amendments, the B.S.S. do tell a rational user, the type of material to expect, when he places an order with a manufacturer.

I would be glad of Dr. Crow's further remarks in this matter, as his two conflicting lines of thought are not reconciled by his concluding analogy, as he advises the user to use convenient specifications with restraint and possibly avoid them. How then will the user know he

is getting phosphor bronze, aluminium, or whatever he has ordered to a reproducible pattern ?

Yours sincerely,

C. G. Williams.

Slough, Bucks.
August 16th, 1950.

The Editor, Metallurgia.

Sir,

We read with great interest Dr. Crow's article " That Specification : Master or Servant ? " and agree whole-heartedly with much that he writes.

We have gathered the impression that Dr. Crow would like to see some specifications widened in scope. That customers should be more co-operative when material is received which just falls short of the speci-fication, by trying it out. Also, that the habit of using the same (say) Brass specification for every press job without due attention to the nature of the pressing should be checked.

Dr. Crow fails however, to state what service he expects a specification to render.

Surely the whole purpose of any standard for metals is : (1) To help the manufacturer to concentrate his production on a few types of material, rather than be forced by competition to break up steady production to cope with large numbers of orders for odd grades and temper ; (2) To ease the designers' problem in choosing a suitable metal.

As such, the specification is a Servant.

There are, however, occasions when a designer just cannot find a specification to fit his job. This is the time to call in the experience of his supplier. Possibly nothing short of a non-standard material will do and brings us to our third point.

The designer enquires the price of the non-standard material and finds to his pleasure, that it is not very different from the nearest standard material and immediately gives up further efforts to fit a standard metal to his design.

It has always been our contention, that the price differences between standard and non-standard alloys laid down by the various associations, are far too small to be economical. Something does not come from nothing and if the small user of an unusual alloy can buy it for little more than a Standard one, it only means the real mass producer is having to pay too much to compensate, thus delaying the benefits that true mass production must bring ultimately.

Dr. Crow particularly mentions Merchants and Stockists, so we may perhaps be allowed to answer that there can hardly be any buyer or supplier—for the Stockist is both—who is more interested in using Standards. He has trouble enough now stocking all the B.S.S. in their various sizes, without adding curiosities of alloys, etc.

May we sum up by saying, that we should have liked to see Dr. Crow's article written more from the angle " If you are a small user, you should make every effort to use a B.S.S., and suppliers will try to reduce their price accordingly. But make sure your designer knows what B.S.S. is nearest to his requirements and then that he alters your job is necessary, so that the B.S.S. will be satisfactory."

We feel it is impossible to lay too much emphasis on the necessity in modern times of using British Standard Specifications.

Yours faithfully,

P. Lovett,
Metallurgist.

Messrs. H. Rollet and Co., Ltd.,
Chesham Place, S.W.1.
July 20th, 1950.

Dr. Crow has advised us that he will reply to corres-pondents, through these columns, in due course.—Editor.

Industrial Finishes Exhibition

formulation of over 350 different raw materials into the standard range of industrial (stoving and air-drying), coach, marine and decorative finishes.

Vitreous Porcelain Enamelling

A large number of vitreous enamelling firms will be represented on the stand of the Vitreous Enamellers' Association (No. 48a). The stand depicts the processes of manufacture and application of vitreous porcelain enamel, both of which involve the complete fusion of the constituent materials and samples of the natural minerals used, such as felspar, quartz, fluorspar, etc., together with a range of metallic oxides used in colouring the glass. A model of a kiln in which the materials are melted together at over 1,200° C. will be shown, and samples of the melted glass lead to a working model of a continuous porcelain enamelling furnace, operating at 750°–850° C. and carrying out the final firing or fusing process. A range of coloured plaques show single and multiple colours, all of which are inorganic and per-manent. The question of good and bad designs of components to be enamelled will also be an important feature, and exhibits of good and bad design and the effects of wrong construction will be illustrated side by side with practical exhibits of the correct procedure. A varied range of exhibits will illustrate the uses of the process and a panel of consultants will be available to discuss all aspects of vitreous porcelain enamelling as applied to iron and steel components.

British Constructional Steelwork Association

The above Association occasionally prepares documents of technical interest to its members and it has recently come to the conclusion that many of these have a much wider field of appeal. It has, therefore, been agreed to make such documents available outside the membership of the Association in the form of brochures which will be issued from time to time. The first of these, which gives examples of Structural steelwork design to con-form with the requirements of the new British Standard 449/1948, " The Use of Structural Steel in Building, " has just been issued. Copies will be made available free of charge to any interested person upon application to the Association at Artillery House, Artillery Row, Westminster, S.W.1.

Recent Developments in Materials, Tools and Equipment

Composite Forgings for Die Manufacture

An excellent example of Anglo-American co-operation in the interests of increased productivity is to be seen in the setting up at Luton of a plant for the production of composite forgings for the manufacture of dies for blanking, trimming and clipping sheet metal in the power press. It owes its existence to an exchange of ideas between one of the largest American automobile manufacturers and a British engineering group, followed by facilities being afforded for investigation and study of the development in the United States.

In this country the present method of building panel trimming, clipping and blanking dies is that which obtained in America prior to the introduction there of composite forgings. For instance, a panel trimming die comprises solid rectangular blocks of tool steel which, after being machined all over, are mounted on an iron casting. They are then dowelled into position to form a complete ring which must again be machined as a unit so that the cutting edge conforms to the contours of the panel to be trimmed. Following this, it is necessary to dismantle the whole die for the purpose of hardening the die steels, which are then re-assembled on the casting, accurately adjusted and finally dowelled into position.

In the new method, the rectangular tool steel blocks are replaced by composite forgings which are homogeneous compound units fashioned out of separate elements and comprising a cutting face of tool steel welded to a wide mild steel base. The tool steel cutting face very closely approximates to the required contours of the finished die in both elevation and plan. A set of composite forgings for a particular die will, therefore, assemble like a jig-saw puzzle into a complete die with a minimum of machining and with the following advantages over the old method :—

1. Due to the fact that the use of composite forgings allows the face of the casting to be maintained flat or, in extreme cases, to have only one or two steps, in contrast with the series of steps necessary to accommodate the relatively shallow tool steel blocks in the old method, a considerable saving in machining time is achieved.

2. As composite forgings are manufactured dimensionally to the specific requirements of a particular die, where a contour is necessary it is provided in the composite itself, which again reduces the machining and fitting time as compared with that necessary for machining solid rectangular blocks.

3. A further saving in time results from the elimination of dowelling into hardened tool steel, as all dowelling is carried out in the soft mild steel base after hardening. With dowelling a final operation, a considerable amount of lapping and grinding, which had to be applied to the cutting edges and to the dowel holes themselves to correct distortion during hardening, can be eliminated.

4. In the event of a breakdown of the die, a replacement composite forging can be supplied within 24 hours and brought rapidly into production. The advantage here is obvious when compared with the weeks so frequently necessary, formerly, to get back into production.

From a study of the composite forging method of die building in America, as compared with the old method used here, it is estimated that a saving of at least 30% of the die-making man hours can be effected in the motor

First stage in die-building. Composites, after machining top and bottom, are being fitted to the assembly casting.
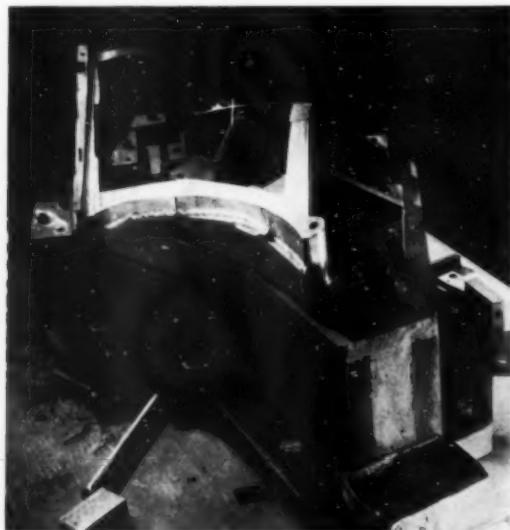
Fully machined composite forgings assembled on to the casting base. Note the piercing hole.

industry alone. Apart from the saving in costs, the car manufacturer can reach flow production on a new model much more quickly, an important factor for the industry's dollar-earning capacity.

Although originating in the automobile industry, the development applies to the whole of the mass produced sheet metal industry.

Composite Forgings, Ltd., Luton, Beds.

New Lincoln Electrodes

AFTER several years' continuous research, Lincoln Electric Company can respond to industry's insistent demand for a quality electrode with the operational characteristics of the popular easy-flow electrode and suitable for use by inexperienced labour. The new product, "Multiweld," runs easily on either AC or DC, shows exceptionally low spatter loss and yet has the Lincoln "Shielded-Arc," good penetration and satisfies the requirements of B.S.S. 639, Lloyds and Board of Trade. It lays down a smooth weld of pleasing appearance and de-slags so easily that in most cases the slag actually peels off as the weld cools. It works well on transformers of low open circuit voltage and runs exceptionally quietly under all conditions, even in the larger sizes. This is chiefly due to the low spatter loss and the stabilised arc and holds true regardless of the ability of the individual operator. The high performance of the $\frac{3}{32}''$ size on light gauge sheet metal means lowered inventory costs in shops where many different gauges are in use. The coating is exceptionally tough and stands up well to handling and storage.

The companion electrode "Positionweld," has precisely the same characteristics but is easier to use in the vertical and overhead positions.

The high rate of deposition will be an important contribution towards lower costs in such industries as shipbuilding, sheet metal and mass production where high speeds with consistent results are essential. Many shops will also gain additional benefit from the smooth finish and complete absence of porosity, which should result in lower finishing costs and less trouble with paint peeling from welds.
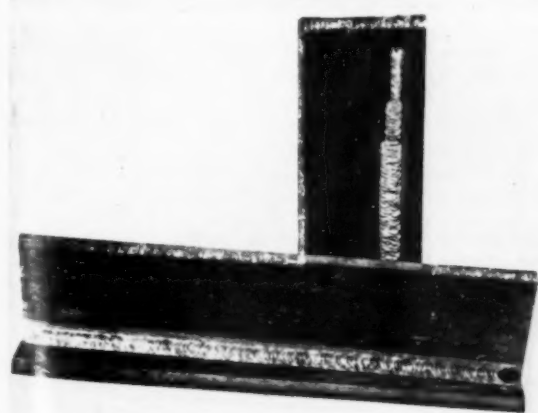
Normal views of Multiweld and Positionweld. The downhand run is with a ⅛ in. electrode and shows the excellent finish obtainable with the larger size—a very important factor in the mass production of consumer goods.

The physical qualities of a specimen prepared to the draft B.S.S. shortly to be adopted as standard are :

Yield Point	27·6 tons/sq. in.
Ultimate Tensile Stress..	34 tons/sq. in.
Elongation	33% on 1·5 in.
Reduction of Area	56%
Izod	52 ft./lb. average

Full operating details are contained in Procedure Sheet Nos. 41 and 42, available on request.

The completion of "Multiweld" and "Positionweld" coincides with the appearance of a new type of moisture-proof soldered container. This, combined with the toughened coating, should ensure their standing up to almost any storage conditions.

Lincoln Electric Co., Ltd., Welwyn Garden City, Herts.

Bunker Level Control

A VARIETY of interesting uses have been reported for the B.L.49 bunker level control introduced recently by Radiovisor Parent, Ltd. This simple unit, which is quickly and easily screwed into position, incorporates a projecting rod-type electrode which may be of any length to suit the requirements of the installation. It is electrically connected to a neat control unit comprising a single-stage thermionic valve amplifier incorporating red and green lights and terminals for connection to an external audible alarm.

One interesting application of this equipment is for controlling the level in bunkers holding coarse, crushed silica stone, the moisture content of which varies up to 15 per cent. In the past, due to inattention on the part of the operators, damage and hold-ups have been caused through failure to stop the feeding mechanism when the bunker was full, and thus the introduction of some form of foolproof and automatic control became imperative. When damp, this material sticks to glass with a tenacity almost equal to that of cement. Consequently, the use of ordinary photo-electric methods involving the projection of a beam of light on to a receiver, is not possible because of obscuration of the glass windows.

The material is stored in a twin bunker fed from a common chute having a two-way outlet which, by movement of a plate valve, can be arranged to discharge into either bunker. Two 12-in. electrodes, one in the top of each bunker, are connected to a common control panel : to avoid chances of damage, the position is chosen so that neither electrode is in the direct line of flow of the material as it discharges into the bunker.

Except when the bunker is full, a green light shows on the control panel, indicating that filling can proceed. However, when the material level rises until it touches the electrode, a circuit is completed, thus closing a relay and causing a red light to show. At the same time, the motor responsible for feeding the material to the chute is automatically stopped, and an audible alarm sounded to attract the attention of the operator.

Although not fitted to this particular plant, the control system could be extended so that, as the material is drawn off and the level falls, it uncovers a second electrode. When this occurs, the motor is restarted to refill the bunker.

A somewhat similar unit is also being installed for controlling the level in bunkers used for the storage of fertilizers. It will be appreciated that the above equipment is equally applicable for controlling liquid levels.

Radiovisor Parent, Ltd., 1, Stanhope St., London, N.W.1.

CURRENT LITERATURE

Book Reviews

THE REFINING OF NON-FERROUS METALS

THE published proceedings of a symposium do not make easy reading. Unlike a textbook written by a single authority there is no easy flow of argument, no connected train of thought, and equally there is no uniform style of writing to which we can get accustomed. Nevertheless, the learned societies are performing a useful function by arranging general discussions on specific subjects from time to time. By dividing such a subject into its several aspects and prevailing on recognised authorities to write papers on each aspect, it is ensured that the most advanced thinking and experience find their way into the published proceedings, at any rate when the symposium is well organised by a representative body. The general discussion can also be of great value if properly stimulated and presented. The present volume is successful in nearly all these respects. It serves the further purpose also, as far as the general metallurgical public is concerned, of " re-asserting the Institution's function to act as a platform for the discussion of metallurgical subjects," to quote from the Introduction by Sir Arthur Smout, chairman of the symposium committee.

In this way, the Institution has largely met the criticism sometimes levelled against its publications of being almost wholly devoted to mining and only slightly to metallurgy. As Mr. W. A. C. Newman, the President, stated, however, at the opening session, the refining of metals is one of the main concerns of the Institution ; and this claim has been staked in unmistakable fashion.

Nineteen papers were presented and discussed during the two-day meeting. They cover an immense field, and the dominating impression they leave on the mind is of the very wide variety of methods used to refine the different metals. Rarely does one find two methods which are similar, ranging as they do from electrolytic parting, cupellation, fire refining, reduction, distillation, and flux refinement to aluminothermic processes. Leading authorities make contributions in each field and there is little doubt but that the volume will serve as a standard work of reference for many years.

Spectrographic control in the refining of metals is covered by Mr. D. M. Smith, who gives an excellent review, of a somewhat elementary character, of the methods used with a few of the commoner metals. Mr. A. R. Powell contributes an excellent and concisely written discussion of the problems encountered in the production of some of the rarer metals. It would be hard to find anyone better informed than Mr. Powell on this most interesting subject, and his paper is a model of its kind. These rare metals, such as germanium, gallium, rhenium, chromium and vanadium, are receiving increasing attention because of their applications in the electronic and high-temperature fields. Mr. A. E. Richards, in his paper on the refining of gold and silver, makes the important point that for these metals the refining process must be rapid because of interest charges. A parcel of gold bullion valued at £1,000,000 incurs interest charges of nearly £100 per day ! This is a most comprehensive and informative paper. The precious metals are considered by Mr. H. Gordon Dale in a nice little paper dealing with the special problems of refining residues and wastes. Mr. H. J. Miller presents a most valuable paper on the fire refining of copper, most carefully, if somewhat ponderously, written. The traditional methods of refining copper receive most attention from Mr. Miller, while the newer induction-melting and arc-melting methods are covered in a very readable contribution by Mr. R. H. Bauld, who also devotes attention to continuous casting. The electrolytic copper refinery of the Rhodesia Copper Refineries, Ltd. at Nkana, Northern Rhodesia, is described by Messrs. W. J. Friggens, Eric W. Page, and Thos. Milligan. This is a wonderfully organised plant, although one or two contributors to the discussion criticised the high anode spacing and low current density in the tank house. These are, however, apparently due to the special local conditions encountered, including the lack of skill, in the early stages, of native labour. Another interesting point raised in the discussion of the papers on copper was the question by Mr. G. L. Bailey as to whether it was really necessary to oxidise remelted cathodes to $0 \cdot 9\%$ oxygen and then pole back to $0 \cdot 03\%$. A not altogether satisfactory explanation is given by the copper refiners. The refining of bismuth is discussed by Mr. A. R. Powell in a useful short paper, and that of cobalt at the Rainham Works of Murex, Ltd. by Mr. P. S. Bryant. Straightforward chemical methods are used for the refining of cobalt from the converter slag produced from the Nkana mine, which fortunately contains only about $0 \cdot 05\%$ nickel. Mr. Frank A. Green gives an excellent account of the well-known process for the refining of lead and associated metals at Port Pirie, South Australia ; while Mr. Stanley Robson speaks from a breadth of experience in his description of the refining of zinc, especially refluxing methods. Mr. E. H. Jones gives a good review of the problems of tin refining, and Mr. T. F. Smeaton deals with the preparation of tungsten for use in electronics and carbide products. The latter is hardly a refining process in the accepted sense, but rather a purification of one or other of the intermediate products from which tungsten is derived.

Mr. G. L. Miller reviews in a comprehensive way the American and German methods for the production of ductile zirconium ; Mr. E. F. Emley describes the flux-refining and other methods of purifying magnesium in an extremely detailed paper ; Mr. J. Waddington examines the refining of aluminium by electrolytic processes and some other less-common thermal methods ; Dr. P. Gross describes the catalytic distillation of aluminium and gives the results of some laboratory experiments on the process ; Mr. T. Burchell contributes an interesting paper on the extremely simple aluminothermic process for the preparation of commercially pure chromium manganese, and special alloys such as ferro-columbium ; and finally Mr. J. A. Blake gives a short description of the processes used for the production

of low-carbon (0·03–0·15% C) ferro-chrome direct from chrome ore.

The discussion is included at the end of each paper or set of similar papers. Most of the papers stimulated good discussions, but it is not surprising that the more specialised papers should have been accepted in comparative silence. The organisation of good discussions is a task requiring much forethought and advance stimulation.

The volume is well edited and produced, with only a few minor technical blemishes, and a most welcome feature is a very comprehensive subject index. The price is, one supposes, not excessive by modern standards, but it is high enough to place it beyond the reach of most of those who should read it, namely, the students of metallurgy.

W. G. ASKEW

PROTECTIVE COATINGS FOR METALS

THE objective of this book is to present information on protective coatings in the light of advances during the last decade, and the authors have attempted to include chemical and physical properties, durability and inspection as well as mode of formation under each type of coating.

The book is subdivided into two main sections covering metallic and non-metallic coatings, and supplemented by appendices on inspection methods and by tables giving summarised information on the principal coatings. The metallic coatings embrace those produced by electrodeposition, by dry processes of hot dipping or cementation and by metal spraying. The non-metallic coatings include those formed by phosphate and oxide film-forming processes, by anodic or immersion processes on aluminium, magnesium and zinc and their alloys. They also cover paints, lacquers, enamels and temporary protectives. The mode of presentation of information on these subjects is novel, the authors having struck a nice balance of theory, practical process detail and inspection data, together with comments upon the properties and applications of each coating. The style is terse and simple, with superfluous descriptive matter rigorously avoided, which explains the mass of facts in so few pages.

Outstanding features calling for comment are the evidence of practical knowledge of industrial finishing, this being shown by the manner in which difficulties are dealt with—e.g., zinc plating of iron castings, the inclusion of proprietary processes classified in their appropriate sections, and the systematic presentation of inspection procedures. Criticisms can only be made of omissions, and they may be regarded within the scope of "suggestions for the improvement of this volume," as requested by the authors in their preface.

Cleaning processes are adequately dealt with, so is the electrodeposition of metals except that more could have been included on copper plating, especially the Rochelle salt, fluoborate, and other modern high speed electrolytes, and on Speculum and precious metal plating, especially gold, silver and rhodium. The latter must be regarded as within the scope of "protective coatings," because they have to fulfil this role when used on copper-rich and aluminium-rich alloys for their properties of high frequency conductance in the radio industry.

Hot-dip processes are well treated for sheet and strip, but more on the treatment of components in tin or in solder, including the bulk hot tinning or soldering of small parts, will be welcomed in the next issue. Metal spraying is briefly presented, and this section can advantageously be expanded, with particular reference to zinc spray for components and structures.

Phosphate processes are admirably presented, this referring to theoretical and practical aspects, and the boldness in including proprietary names is commended. The same applies to oxide film-forming processes, and to processes for aluminium, magnesium and zinc.

Paints, lacquers and enamels are well discussed, and this section lends itself to expansion to include detailed sections on the finishing of components and structures, giving schemes for the various metals, and including "tinplate printing." No reference is made to the phosphoric acid type "etch primers."

Temporary protectives are comprehensively covered and well classified; future issues could advantageously include the polyvinyl resin coating such as "Birlon" for metal sheet, strip, section, etc., and "Cocoon" for spraying over completed equipments.

The segregation of inspection tests into appendices has enabled them to be presented compactly yet informatively and without undue repetition. The summarised data in tables for all the outstanding finishes dealt with, are commended, as they provide much useful "at-a-glance" information. Over 200 references are given to original papers, wisely grouped together at the end of the book, and the reference numbers are boldly given in the text.

The authors are to be congratulated on producing a book on Protective Coatings for Metals, in such a style, firstly that it does not clash with volumes already existing, and secondly that it lends itself to revision at regular intervals "in the light of advances made in this field during that interval." Their method of blending theory and practice for each coating dealt with, the theory in simple but factual terms, and the practice including accurate formulæ, practical comments, and assessment by inspection, is much to be admired. The book is short and easily readable despite the mass of scientific and technical facts, and the omissions from the first issue, which it is the duty of the reviewer to note, do not detract in any way from its merits.

E. E. HALLS

SPECIFICATIONS FOR HEAVY DUTY ELECTRIC OVERHEAD TRAVELLING CRANES FOR USE IN IRON AND STEEL WORKS

THIS specification is the outcome of the setting up by B.I.S.R.A., in 1945, of a Steelworks' Cranes Sub-Committee, under the chairmanship of Mr. W. F. Cartwright, Managing Director of the Steel Company of Wales. Since 1947, the Sub-Committee has included representatives of the crane manufacturers, as well as the iron and steel industry. Although drawn up by and for the iron and steel industry, it is likely that the specifications will be of interest to users in a much wider field.

In the past, many designs of steelworks electric overhead travelling crane have failed to stand up to the severe duties and handling they have received. The

result has been the evolution of the modern heavy-duty steelworks crane, and this development has now reached a stage when some measure of standardisation is desirable.

The specification of performance in terms of accelerations and speeds by the buyer becomes desirable because it is seldom possible in an iron or steel works to specify an exact duty cycle. For cross travel motion, therefore, three, and for long travel motion five, performance classes have been detailed on which specification by the purchaser may be based. These classes take into account both the initial acceleration and the speed of the motions. The investigation from which these data were derived were reported in the *Journal of the Iron and Steel Institute* 1950, Vol. 164, April, p. 439.

As a guide to the purchaser there is an appendix " Information to be supplied with Enquiry," with a corresponding appendix for the manufacturer, " Information to be supplied with Tender," both cross reference to the specification clauses. A third appendix sets out the various standards and regulations referred to in the specifications, and a fourth summarises the safety regulations for steelworks cranes.

The British Standards Institution has been consulted from the inception of this project, and intends to use the specification as a basis for a British Standard in the light of the experience that will be gained in its operation during the next year or two.

RECOMMENDATIONS FOR THE DESIGN AND FABRICATION OF ARC WELDED STRUCTURAL STEELWORK, BUILT-UP GIRDERS AND COMPRESSION MEMBERS.

Publication T26 of the British Welding Research Association, 29, Park Crescent, London, W.1., 12 pp., 1s 6d.

THIS Memorandum is the second* in the series which is based on the work of the FE.13 Committee of the British Welding Research Association on Codes of Practice for Welded Building Structures, and which is intended to provide guidance on the design, fabrication and erection of arc welded structural steelwork. The recommendations have been drawn up after a detailed study of existing practice. Research continues to be carried out on the subject.

* The first, entitled " Stanchions, Bases, Caps and Joints," is available in the Technique and Memorandum Series, reference T.17, price 9d.

CONFERENCE ON FOUNDRY STEEL MELTING.

Published by the British Iron and Steel Research Association, May, 1950, 73 pp., 10s. net.

THE Steel Castings Divisions of B.I.S.R.A. has published the " Proceedings " of its fourth conference, held in September, 1949, which had the general theme of foundry steel melting. The papers presented were spread over four groups :—

Cupola/Converter Process—The Scope and Limitations of the Cupola/Side-Blown Converter Process; Side-Blown Converter Design; The Aerodynamical Aspects of Side-Blown Converters; The Control of Sulphur and Phosphorus in the Cupola/Converter Process; The Application of Oxygen Enrichment in the Cupola/Converter Process.

Electric Arc Process—The Specialised Control of the Electric Arc Process for Foundry Use; Refractories for Electric Arc Furnaces; Electrical Characteristics, Output and Design of Arc Furnaces.

Other Melting Processes—The Use of the Open Hearth Process in Foundries; Foreign Steel Foundry Melting Practice; The Use of the High Frequency Process in Foundries; The Design of the High Frequency Furnace. *General*—Steel Foundry Productivity; The Fluidity of Different Steels; The Cost of Melting in Foundries.

The discussions on the various groups of papers, as reported in the " Proceedings " give an interesting indication of some of the topics exercising the Steel Foundry Industry at the present time.

" WIGGIN NICKEL ALLOYS "—NO. 326.

THE current issue of " Wiggin Nickel Alloys " includes short illustrated descriptions of some of the many uses of Monel and other nickel alloys in steam power plant. Other interesting items deal with Monel fishing reels, aquarists equipment, water baths, cinema resistances for electrical dimming equipment, a new gas-fired space heater and new ceramic-to-metal seals for use in electrical and radio apparatus.

High nickel alloys are employed for a variety of purposes in the equipment described and this issue of " Wiggin Nickel Alloys " will have a wide appeal to readers. Copies of this publication may be obtained, free of charge, upon application to :—Henry Wiggin & Company, Limited, Wiggin Street, Birmingham, 16.

Trade Publication

One of the most attractive catalogues received is that from Exors. of James Mills Ltd., Bredbury Steel Works, Stockport, Cheshire, which has been specially produced as a Centenary Edition. The history and growth of this progressive firm, since its inception in 1850, is briefly outlined in an informative Foreword. From small beginnings this firm has grown steadily and to-day schemes for new extensions and plant renewal are in progress which will increase the area of the works from 337,000 sq. ft. in 1949 to almost half a million sq. ft. by the end of this year.

With a production capacity of about 2,500 tons of bright steel bars a week and as supplies to every type of engineering trade, this firm is the largest maker outside the United States. The range of sizes covers rounds from $\frac{1}{16}$ in. to 6 in. dia., squares from $\frac{1}{4}$ in. to $3\frac{3}{4}$ in. square, hexagons up to 3·550 in. across the flat, over 1,200 flat sizes and more than 1,600 special shapes most of them in a wide range of qualities. All these items are made at Bredbury from raw material rolled in the firm's works and carefully checked by laboratory staff and technicians at all stages of manufacture. In the sphere of keys, pins, cotters and railway permanent way fastenings, the firm's extensive experience and years of production enable it to give exactly what is required either to standard specifications or to customers' own requirements.

This 68-page booklet not only gives information concerning the products of this Company, including the readily machinable " Ledloy " steels, but also includes a useful glossary of technical terms and much tabulated data of considerable value, especially for reference purposes. The booklet is admirably produced and well bound in board covers and it will be invaluable to all users of steel.

METALLURGICAL DIGEST

Nodular Gray Iron Compared with other Cast-Ferrous Metals

By G. Vennerholm, H. Bogart and R. Melmoth

THE production of nodular iron is generally accomplished by a two-stage operation. The first stage is that of the addition of a small amount of cerium, magnesium, lithium, or similar carbide formers which promote the formation of white iron in an iron normally solidifying gray. The second stage is an opposing stage in which a ferrosilicon type of inoculant overcomes the tendency towards white iron, causing the graphite to precipitate out in the form of small spherulites. In general, it can be stated that any iron which, including its ferrosilicon inoculant, solidifies gray can be made nodular with magnesium.

The choice of carbide stabilising agent is governed by economic considerations which seem to favour the use of magnesium. In addition, limitations surrounding cerium and some of the other metals, such as the requirement that the iron in question be hypereutectic, do not apply to magnesium. The magnesium can be introduced as a relatively pure metal, as a non-ferrous alloy including magnesium, or as a ferrosilicon modified with magnesium. It is obvious that with a boiling point of about 1,110° C., pure magnesium becomes gaseous at molten iron temperatures and reacts with explosive force in the presence of air. Similarly, the magnesium-rich alloys are more violent than those containing smaller amounts of magnesium.

Numerous alloys have been investigated but most of these have been discarded in favour of magnesium-nickel, magnesium-copper, and of late, magnesium-copper ferrosilicon. The latter is of particular interest in that this alloy does not involve addition of large amounts of nickel or copper.

The relative amount of magnesium required to produce nodular iron depends upon the amount of sulphur present in that the first reaction, following the addition, appears to be the formation of magnesium sulphide, thereby removing the sulphur from the field of action. The reaction proceeds until the sulphur analysis approximates 0·02%, at which point the magnesium becomes effective in changing the form of the graphite.

Experience has shown that a minimum of 0·035% magnesium must be present in order to effectively produce completely nodular iron in a typical cupola analysis. If the recovery exceeds 0·1% magnesium, however, excessive carbide stabilisation occurs, ultimately resulting in a stable white iron. Table I shows the approximate recovery in per cent. that can be expected from various magnesium-copper and magnesium-copper-silicon alloys when added to an iron containing 0·03% sulphur in amounts sufficient to produce nodular iron. These figures are representative only and should not be used as a yardstick. Efficiency of magnesium recovery is influenced by

TABLE I.—RELATIVE EFFICIENCY OF MAGNESIUM ADDITION AGENTS

(Based on Treatment of 0·03% S iron at 1,480° C. to give 0·08% Residual Magnesium)

Alloy	Mg. Addition	% Mg. Recovery	Alloy Wt. Ton
6% Mg.-10% Cu-Fe-Si[1]	0·3%	32%	100 lb.
13% Mg.-10% Cu-Fe-Si[1]	0·4%	24%	61 lb.
20% Mg.-80% Cu[1] ..	0·53%	19%	53 lb.
50% Mg.-50% Cu[2] ..	0·7%	14%	28 lb.

1 Added to stream at 1,480° C.
2 Added to covered ladle at 1,480° C.

several factors which must be carefully controlled if consistent results are to be expected.

The temperature of the molten iron has a considerable bearing on the relative magnesium recovery and, consequently, on the economy of the operation. The lower the temperature consistent with safe operation, the greater is the recovery. Whereas in Table I the recovery from a 6% magnesium-copper-ferrosilicon alloy is about 32% at 1,480° C., the expected recovery at 1,370° C., if such a temperature could be used safely, is approximately 45%. The chilling effect of the magnesium alloy and the inoculant necessitates treating the base metal at a higher temperature than that required for the satisfactory pouring of the casting.

This would seem to favour the magnesium-rich alloys such as 50% magnesium — 50% copper, where smaller quantities of the carrier element are required. The increased volatility of these alloys, however, with the resultant safety hazard, more than offsets this advantage.

The source of the molten metal, whether blast furnace, cupola, or electric furnace, does not seem to have any appreciable influence on the casting or engineering properties of the nodular

TABLE II.—PROPERTIES OF NODULAR IRON AND OTHER FERROUS METALS

Property	Gray Iron S.A.E. 111	Alloy Iron Ni, 0·80 Cr, 0·20 Mo, 1·00	Malleable S.A.E. 35918	Pearlitic Malleable S.A.E. 43010	Steel S.A.E. 0030	Steel S.A.E. 0105	Nodular Iron High Strength	Nodular Iron High Ductility (as cast)	Nodular Iron High Ductility (Annealed)
Yield Point, lb./sq. in.	—	—	25,000	43,000	35,000	85,000	80,000	55,000	45,000
Tensile Strength, lb./sq. in.	30,000	70,000	53,000	60,000	65,000	105,000	100,000	80,000	65,000
Elongation, %	—	—	18	10	24	17	1·5	10	16
Reduction in Area, %	—	—	—	—	35	35	1·0	8	20
Brinell Hardness	190	286	130	170	130	217	255	190	160
Modulus of Elasticity	19×10^6	21×10^6	25×10^6	25×10^6	30×10^6	30×10^6	24×10^6	24×10^6	24×10^6
Impact Ft.-lb., 0·798 in. dia. bar..	10	20	—	—	—	—	40	180	260
Type Melting Unit	Cupola	Cupola	Cup.-El.	Cup.-El.	E. Fce.	E. Fce. Quench & Temp.	Cupola	Cupola	Cupola
Heat Treatment	None	None	Anneal	Anneal	Anneal		None	None	Anneal
Casting Yield, %	80	75	52	50	50	50	70	70	70
Metal Cost per ton Castings:									
Raw Material	840·81	870·73	840·40	840·95	831·85	846·84	859·64	863·86	863·86
Conversion	6·75	7·20	14·69	15·18	31·83	31·83	7·71	8·65	8·65
Heat Treating	—	—	9·00	9·00	9·00	18·09	—	—	6·00

iron family. This does not mean that the properties are independent of the chemistry, for it must be emphasised that definite chemical limitations exist, especially in the manufacture of the more ductile forms of iron. The appreciable sulphur pick-up associated with cupola operations is a distinct disadvantage in that most of the sulphur must be eliminated before the magnesium treatment becomes effective in re-shaping the graphite. This can be done by conventional desulphurising methods or by the use of additional amounts of magnesium alloy above that required for carbide stabilisation. Foundries equipped with electric furnaces may find it an advantage to use these units instead, as then a base iron of initially low sulphur, at controlled temperatures, can be produced. Regardless of which unit is used for the manufacture of nodular iron, a thorough understanding of the effect of the various elements on resultant properties is necessary in order to determine the proper chemistry to meet physical requirements.

In order to critically evaluate the importance of nodular iron, it may be helpful to study Table II which tabulates the physical properties representative of various cast ferrous alloys and some figures, given in dollars, pertaining to their relative cost of manufacture.

Applications

The potential uses to which nodular iron might be applicable, on the basis of engineering properties, are suggested in four groups. The first of these is that entire field where a non-flaky graphite in a selected matrix has proved so successful. This would place nodular iron in direct competition with the various types of malleable irons. There is, however, no immediate economic advantage in replacing present malleable iron castings with nodular iron, but its early application may be associated with section sizes larger than present malleable iron casting practice. The second field where this material might find application is in competition with conventional gray iron castings solely on the basis of improved engineering properties, except in the case of alloy cast irons where an economic advantage is indicated. A third field is the possibility of its use instead of steel castings where the castability or machinability of the part in question might dictate its consideration. Finally, there is evidence that nodular iron can be hot worked in the neighbourhood of 927° C. with good results.

High Temperature Steels in Steam Plant Practice

By J. Glen, B.Sc., A.R.T.C., F.I.M.

DURING the recent war spectacular strides were made in the development of austenitic steels and other alloys for use at very high temperatures and stresses. The result of this achievement can be seen in the proved reliability of the modern gas turbine aero engine. Less spectacular has been the great progress made in steels for steam power plant. Since 1920 a revolution has taken place quietly and without fuss to which both the metallurgist and engineer have contributed with immediate and very considerable benefit to the country at large. From that year steam power for electric power generation has been raised four or five times, not as a general practice, but successfully, and in the same time steam temperature has been raised by over 200° F.

Ordinary carbon steels, in which the essential constituents are carbon, manganese and silicon, still form by far the greatest bulk of steels used in steam generating plant. Of these a 0·4% carbon steel is not so good as a low-carbon steel at temperatures above about 820° F. At room temperatures, however, the working stress is usually taken to be one-quarter of the tensile strength. Thus, at lower working temperatures, a high-carbon steel can withstand higher stress than low-carbon steel. The effect of manganese is even more important, since very low manganese steel has very poor creep properties. Fortunately, nearly all carbon steels contain approximately 0·5% manganese, and this amount gives a creep rate of 4×10^{-6} in./hour compared with 40×10^{-6} in./hour for a 0·3% manganese steel each containing 0·12% carbon, tested at 450° C. and a stress of 8 tons/in.², the creep rate at 5 days being taken as the criterion of strength.

From extensive experimental work it has been found that there is only one element which can markedly increase the creep resistance when added to carbon steel. This element is molybdenum. The effect of molybdenum on the creep strength of carbon steel is rather like manganese in that the addition of 0·5% molybdenum gives about 80% of the creep resistance of a much higher molybdenum content. If

the molybdenum content is much below 0·4% the creep resistance rapidly decreases. For this reason a 0·5 molybdenum steel has become standard for those applications where carbon steel has not got sufficient strength. As with carbon steel, increasing the carbon content of 0·5% molybdenum steel tends to decrease the creep resistance.

The other alloy steels used at high temperature are all based primarily on the 0·5% molybdenum composition with the addition of one or more of other alloying elements. Chromium is the main addition to a whole series of such steels, because with increasing chromium content the scaling resistance at high temperature is progressively improved. If the chromium content, however, is much above 1%, the creep resistance of the steel is reduced as compared with the 0·5% steel. Since the problem of scaling has not yet been the limiting factor in design, the only chromium steel which has found favour is one containing approximately 1% chromium. With this amount of chromium the creep resistance is improved as compared with 0·5% molybdenum steel. This improvement results primarily from the fact that the chromium-molybdenum steel does not deteriorate so rapidly.

This chromium-molybdenum steel is gradually replacing the 0·5% molybdenum steel in high-temperature steam plant. Apart from the increase in creep resistance there are two other reasons for this choice. Since chromium stabilises the carbide of the steel, it is found to be an additional safeguard against the possibility of graphitisation, and a chromium-molybdenum steel appears to be less liable to fail with a low elongation.

Up to about 950° F. a molybdenum steel or a chromium-molybdenum steel can withstand a fairly high stress, but above that temperature the stress-carrying ability rapidly falls to a low level. However, the addition of approximately 0·25% vanadium to a 0·5% molybdenum steel increases the creep resistance markedly. Thus, a molybdenum-vanadium steel in the normalised and tempered condition can withstand three times as high a stress as a 0·5% molybdenum steel in the temperature range of 1,000–1,050° F.

A Laboratory Arc-Furnace for Melting Alloys Containing the Refractory Transition Metals

By G. A. Geach, M.Sc., Ph.D., F.I.M. and
D. Summers-Smith, B.Sc., A.R.T.C

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INTEREST in alloys of the refractory transition elements, such as titanium, is at this moment intense, but there are often great, if not insuperable, difficulties in preparing them without serious contamination by conventional methods of melting or powder metallurgy. Difficulties arise in providing both the means of heating the metals to the high temperatures commonly involved and the inert materials required to contain them. Consequently the type of arc furnace in which small melts can be easily prepared without either of these troubles intruding is now of great importance. This justifies a rather more detailed account of the construction and operation of such a furnace than is at present readily available.

In the furnace to be described the specimen, resting upon a water-cooled copper hearth, is the positive pole of a heavy current D.C. arc. The material melts, all except a thin layer near the hearth which protects it from copper contamination : this layer is itself melted later after turning the specimen over. An inert atmosphere of purified argon protects the charge.

Fifty grams of tungsten may be melted in such a furnace in a few minutes, alloys of uranium or titanium are obtained untarnished and virtually uncontaminated, whilst malleable zirconium remains perfectly malleable after melting. It is quite simple to prepare, in one day, 10 or 15 melts of what have hitherto been the most difficult of alloys to obtain.

The Development of Laboratory Arc-Furnaces

Several arc furnaces are already described in the literature, but full accounts are generally of apparatus of a different type from that now to be described.

The first description of an arc furnace was published in 1840 by Hare.[1] This furnace consisted of two charcoal electrodes in an air-tight glass bell. The lower electrode, which was a block carrying the specimen to be melted, could be raised or lowered by means of a copper rod, which passed through a stuffing-box. The top electrode was attached through a rubber bulb which allowed it considerable freedom of movement. Operation *in vacuo* or with a hydrogen atmosphere was possible, power being obtained from batteries of cells which, momentarily, developed up to 30 amperes at 125 volts. In this apparatus Hare reduced calcium phosphate to metallic calcium.

A furnace of consumable electrode type was first used by von Bolton[2] in 1903. This furnace was used for melting tantalum. A rod made of pressed tantalum powder formed the positive electrode of an arc, the other electrode being a water-cooled table onto which the molten tantalum fell. Melting was carried out in a large " evacuated " glass globe immersed in water.

In 1910, Weiss and Stimmelmayr[3] prepared small ingots of tungsten weighing 2–5 grams by striking an arc between two horizontal rods of sintered tungsten, when the end of the positive electrode melted and dripped into a zirconia crucible. A D.C. arc of 110 amperes was used and experiments were carried out in air, nitrogen and hydrogen at 15 mm. pressure.

In 1923 Moore[4] described an arc furnace in which uranium was melted in argon at pressures below 0.1 mm. The uranium powder was pressed into small discs (about 3cm. dia. × 0.5 cm. thick), which were first sintered. One disc was then attached to an upper movable electrode and the other, placed on a water-cooled table of " Monel," was melted by striking an arc between it and the upper electrode.

A more flexible furnace of this type was constructed by Kroll[5] for melting titanium and zirconium. The inert electrode consisted of a tungsten rod connected to the furnace vessel by an arrangement of metal bellows which allowed it considerable freedom of movement during operation. The lower electrode consisted of a water-cooled copper or tungsten-clad copper hearth on which the specimen to be melted was placed. The D.C. arc, run in an atmosphere of 5 mm. of hydrogen or 50 mm. of argon, took 100 amperes at 35 volts.

More recently Parke and Ham[6] have described a furnace capable of producing ingots of molybdenum up to 25 lb. in weight. In this furnace the top electrode was of molybdenum powder which had been sintered to form a rod about 1 in. diameter, the other electrode being a water-cooled copper mould. The top electrode, when the end began to melt after striking the arc, was advanced automatically to maintain a constant arc length. The molten molybdenum was chilled instantly and did not attack the copper mould. Melting was *in vacuo* using 1,000–1,200 amperes, at about 20 volts A.C. In a modified version of this furnace a hopper of powdered molybdenum was included in the furnace vessel and the

1 Hare. A brief Exposition on the Science of Mechanical Electricity or Electricity Proper. Philadelphia, 1840, and *Trans. Amer. Phil. Soc.*, 1841, VII (New Series) 53. Described by C. A. Doremus in *Trans. Amer. Electro-chem. Soc.*, 1908, **13**, 347.

2 W. von Bolton. *Zeits.f. Elektrochem*, 1905, **11**, 45.
3 L. Weiss. *Zeits.f. anorg.Chem.*, 1910, **65**, 279.
4 R. W. Moore. *Trans. Electrochem. Soc.*, 1923, **43**, 317.
5 W. Kroll. *Trans. Electrochem. Soc.*, 1940, **78**, 35.
6 R. M. Parke and J. L. Ham. *Metals Tech.*, 1946, **13** (6). Tech. Publ. No. 2052.
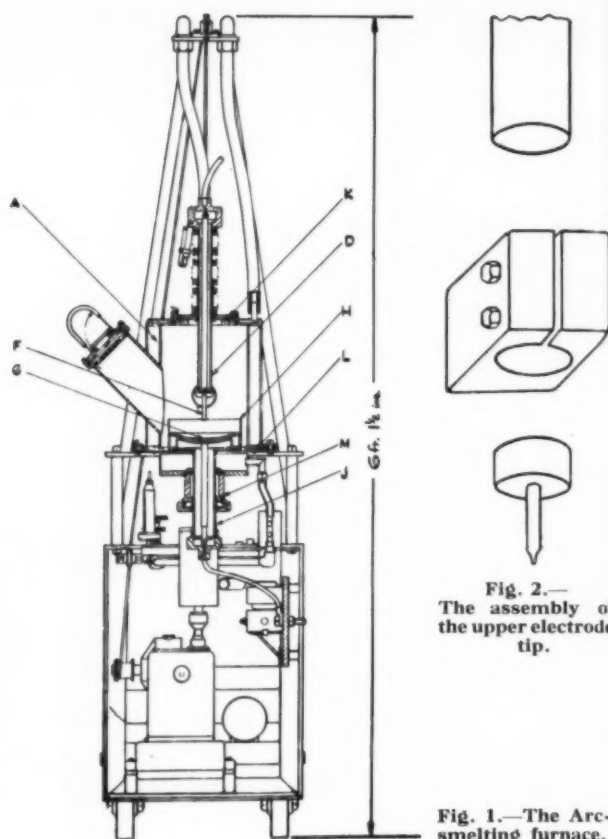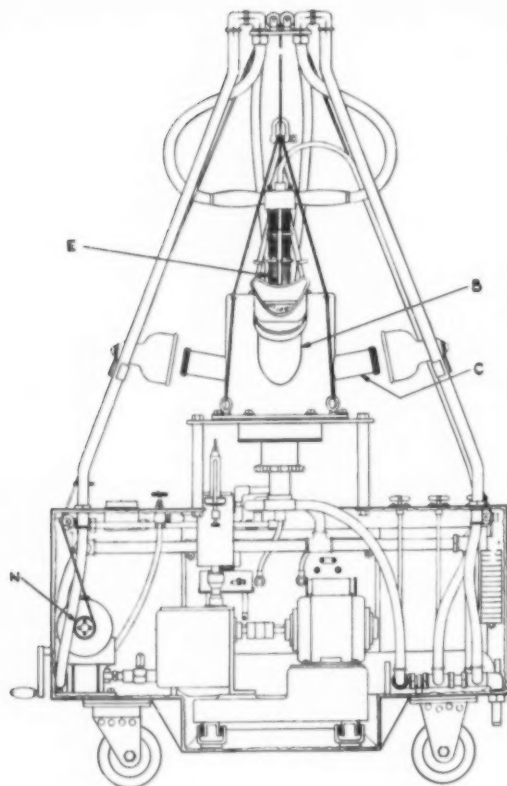
Fig. 2.—
The assembly of
the upper electrode
tip.

Fig. 1.—The Arc-
smelting furnace.

consumable electrode was pressed and sintered continuously from the molybdenum powder.

Parke and Ham also melted tungsten and molybdenum base alloys by making up suitable electrodes. The same method was used by Kessler and Hansen[7] for preparing molybdenum-chromium alloys, the electrode in this case being melted in a continuous stream of argon at atmospheric pressure.

Arc furnaces having a movable tungsten electrode and a water-cooled copper hearth are also in use at the Battelle Institute[8],[9], at Melbourne University,[10] and at the Massachusetts Institute of Technology, where Schramm, Gordon and Kaufmann[11], have prepared uranium-tantalum and uranium-tungsten alloys by using tantalum and tungsten respectively as the inert electrode material ; copper pick-up was negligible. In this case a helium atmosphere was used and the power consumption was 200–700 amperes, at 15–20 volts D.C.

Description of the A.E.I. furnace

The furnace vessel consists of a water-jacketed brass bell A (Fig. 1) with an inside diameter of 10 inches and a height of 12 inches. This rests on a heavy steel base from which it is insulated by a washer of tropical " Bakelite " paper board : a high-vacuum type seal is

7 H. D. Kessler and M. Hansen. Amer. Soc. Metals preprint, 1949, No. 33.
8 S. A. Herres and J. A. Davies. *Steel*, 1949, **124**, 82.
9 O. W. Simmons, C. T. Greenridge and L. W. Eastwood. U.S. Navy Dept. Symposium on Titanium, 1949, 77.
10 R. H. Myers. Personal communication.
11 C. H. Schramm, P. Gordon and A. R. Kaufmann. *Trans. Amer. Inst. Met. Engrs.*, 1950, **188**, 195. (*J. Metals*, Jan., 1950.)

made between the bell and base using a ring of $\frac{1}{4}$-in square rubber.

The hearth G is of $\frac{1}{2}$-in. thick copper, $6\frac{1}{4}$ in. diameter and slightly dished, but having a flat rim about $\frac{5}{8}$ in. wide. It is enclosed by a steel wall H and the lower surface is water cooled. Originally, the cylindrical stem J of this hearth passed through a modified Wilson seal M which allowed some vertical adjustment, but experience proved this to be unnecessary and a rigid seal between a flange on the stem J and the base of the vessel was substituted.

The water-cooled stem of the upper electrode passes through the top of the vessel to which it is linked by an assembly of three extra-flexible ' Hydroflex ' metal bellows. This arrangement allows completely free manipulation of the electrode tip over the whole hearth. A counterpoise connected by cable to the top of this electrode neutralises part of the atmospheric loading when the internal pressure is reduced. Manoeuvring the electrode by the two handles is quite a simple operation. This electrode assembly is electrically insulated from the bell by mica, and from the counterpoise and frame by a porcelain insulator. The tungsten tip of the electrode is hard-soldered to a copper block which is clamped to the water-cooled stem in a simple manner (Fig. 2), allowing rapid replacement.

Satisfactory stable arcs have been obtained with tungsten tips from $\frac{1}{8}$–$\frac{3}{8}$ in. diameter. Thinner tips are suitable when melting small samples or when the current required is small. With larger samples, or currents of

the order of 300 amperes, or more, it is necessary to use the largest diameter tip in order to avoid over-heating and melting the tungsten. With a $\frac{1}{8}$ in. tip, the best striking conditions are with an open circuit voltage of 90 volts and a current of 40–60 amperes. With a $\frac{3}{8}$ in. tip, however, it is necessary to increase the current to 100–150 amperes, but if a low rate of heating is required this can be reduced once the arc has formed. Further control of the heat input during running can be obtained by altering the length of the arc and by changing the voltage of the generator. The potential drop across the arc when the furnace is in operation is normally about 15 volts. The tungsten tip is always the negative pole. Three tubes are brazed to ports in the furnace vessel. The largest B (Fig. 1) is a sighting tube through which the operator can observe what is happening in the furnace. A small prism and lens device fitted to a corner of the observation window projects an image of the arc and hot specimen on to a screen so that several other people may see how the melt proceeds. One of the smaller side tubes C is used for illuminating the hearth from a high intensity lamp before the arc is struck. Probes for further manipulation of specimens, or electrical leads taken into the furnace, pass through the other.

The whole furnace is assembled on a frame which also carries the Metro-Vick DR–1 pump, the argon cylinder and the vacuum valves, and gauges. A small winch N is fitted for lifting the bell to give access to the hearth. The pipes carrying the cooling water also form the supports for the overhead pulley platform.

Power is supplied by a Lincoln " S.A.E. 300 " arc-welding generator capable of giving currents of from 40–400 amperes, and open-circuit voltages continuously variable from 50–100 volts.

Operation of the Furnace

The furnace is evacuated by means of the rotary pump and commercial argon admitted. A suitable pressure is about 20–25 cm., below which there may be difficulty in maintaining a stable arc. Before melting certain metals, for example zirconium and titanium, it is desirable to remove traces of other gases from the argon. This can readily be done by first melting an extra piece of suitable metal to " getter " or take up such gases; zirconium is commonly used. When the current has been switched off this " getter " may be moved by means of the electrode tip, on to the rim around the hearth. Specimens can then be brought in turn from this rim to the centre of the hearth where they are melted, and then pushed back to the edge. Five or six samples can be melted in this way without opening the furnace.

A straightforward melt, as of a piece of sintered tungsten bar, would be made by lightly touching a corner of the specimen with the electrode tip, switching on the current and carefully breaking the contact and maintaining an arc length of about $\frac{3}{8}$–$\frac{1}{2}$ in. A specimen weighing 50 grams melts in two minutes using 250 amperes and, except for the thin layer near the copper hearth, the whole specimen can be kept molten by slowly moving the arc over its surface. After cooling, specimens are quite free from the hearth and can be turned over with the electrode tip, the current, of course, being off. The resulting tungsten bead would be smooth and bright.

Various quite simple methods of preparing alloys have proved satisfactory. For example, alloys of molybdenum and tantalum may be prepared from pieces of previously melted metals, or from metal powders. In the latter
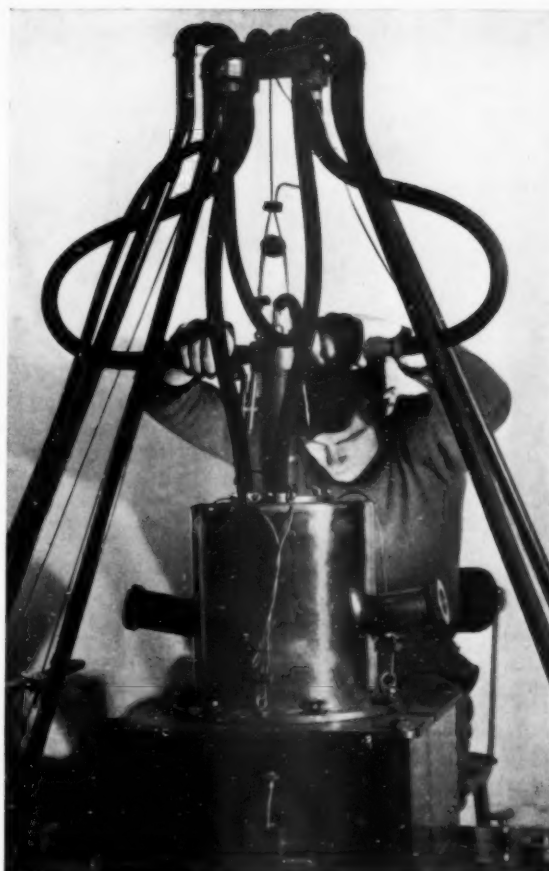

Fig. 3.—The furnace in operation.

case it is usual to pre-sinter the powders in a normal furnace to obtain fritted pieces for use in the arc furnace. This also removes most of the gases in the powders and avoids spattering during the melting. With such precautions the loss in weight during alloying can be negligible. The arc causes great turbulence as it moves over the molten metal and it is usually sufficient to turn over and remelt the specimen twice to obtain perfect homogeneity.

It is convenient to melt one metal of a desired alloy—that of higher melting-point, unless the proportion of this is small—and then to allow this to run down the hearth on to the second metal.

Although, whenever possible, metal powders are sintered and degassed before they are put into the arc-furnace, it is quite possible to melt pressed compacts of metal powders which have not had this treatment. In such a case, immediately the arc has been struck it is drawn out to a length of 1–$1\frac{1}{2}$ in. and the current reduced to 50–60 amperes. The specimen then heats more slowly and uniformly. When the danger of spattering is over, the length of the arc is reduced and the current increased to melt the specimen.

When melted in the arc-furnace, certain metals may give off enough gas to tarnish the specimen as it cools. Uranium is a notoriously troublesome material to melt cleanly but with precautions it can be melted and left
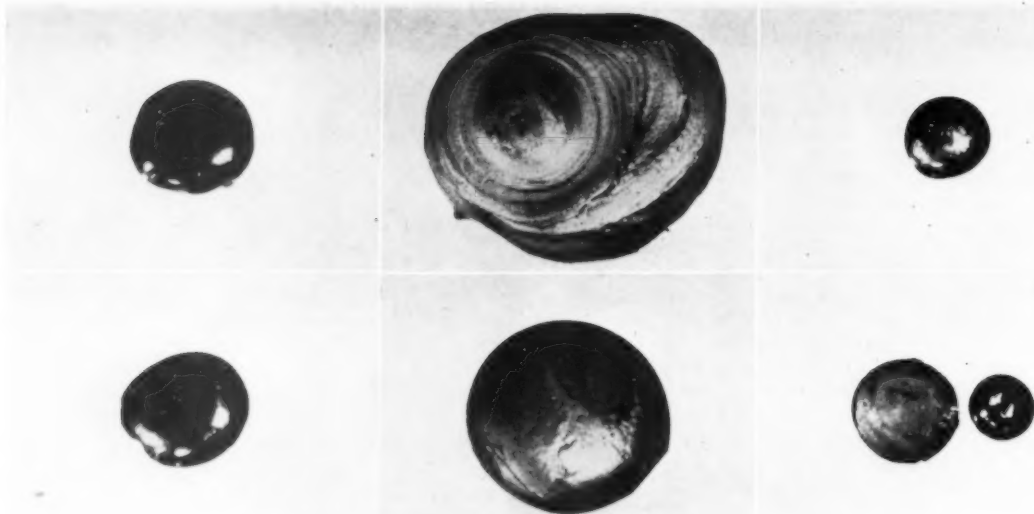
Top (left to right) - Tungsten (3,350°C), Molybdenum (2,625°C), Tantalum Carbide (3,900°C)
Bottom (left to right) - Tantalum (3,000°C), 60% Molybdenum-Nickel, Zirconium (1900°C)

Fig. 4.—Typical samples after melting : magnification × 1. The tungsten weighed 30 grms. and the molybdenum, which is about as large a piece of this material as could be melted, weighed 110 grms. The two zirconium pellets were originally identical but one was compressed to demonstrate that it remained malleable.

with a clean grey surface. To achieve this a tantalum spiral inside the furnace vessel is heated to about 1,400° throughout the melting. Gases given off by the uranium, as it melts, are absorbed and retained by the tantalum. The last traces of oxide are driven from the uranium by raising its final temperature very considerably.

The most serious limitation to the application of this furnace is that imposed by the volatility of certain metals. This not only makes it difficult to attain selected compositions but also to obtain a homogeneous sample or, in an extreme case, such as an attempt to alloy manganese with molybdenum, even to melt the charge. Manganese, of course, is generally hard to alloy but at the temperatures reached in the arc furnace many other metals are significantly volatile.

A related difficulty arises from the decomposition of many carbides when attempts are made to melt them. Tungsten carbide (WC) and tantalum carbide (TaC) melt, but give off such large quantities of fine carbon that the arc may spread to them and it is necessary to open up and clean the furnace. It appears that the carbon of mild steel is similarly driven off.

Rough indications of melting temperatures have been obtained using an optical pyrometer through the viewing window. Considerable absorption of radiation must take place in the silica window and the argon atmosphere, particularly at high temperatures and, of course, the radiation conditions are far from the ideal black-body case. However, by comparison with the melting points of suitable standards, which have been included in the furnace, and can be melted without altering the conditions, a correction can be made. A molten pool of metal is maintained on the top of the specimen and the temperature of the junction of this pool and the solid metal measured ; this temperature will lie somewhere between the liquidus and solidus. Using this method, reproducible " melting-points " of molybdenum-tantalum alloys have been obtained.

Contamination of arc-melted specimens might be anticipated from the hearth, the tungsten electrode or from other gases in the argon, but none has been detected. The last of these sources of impurity is removed by the " getter," as experiments with zirconium,whose mechanical properties are exceedingly sensitive to the presence of small amounts of nitrogen and oxygen, show ; the malleability of van Arkel zirconium is not lost on melting in the arc furnace.

As the upper electrode is negative, any transfer of metal would be from the charge to this electrode and the only danger of contamination in this case is of the metal forming a low melting alloy with tungsten so that drops fall from the end of the electrode into the molten specimen. This would be observed by the operator and should the difficulty arise the tungsten tip could be replaced by some other high-melting metal, preferably one of those in the alloy.

Samples of " spectroscopically pure " nickel, molybdenum and tantalum have been repeatedly melted and compared with unmelted specimens. The maximum amounts of tungsten and copper picked up were less than 0.01% and 0.001% respectively.

Acknowledgments

The Precipitation of Silver Chloride from Perchloric Acid Solutions

By E. C. Mills, A.R.I.C. and S. E. Hermon

Research Laboratories, High Duty Alloys, Ltd.

Some experiments on the separation of small amounts of silver as silver chloride from perchloric acid solutions are described. Within certain concentration limits the precipitation may be used quantitatively even in the presence of nitric or sulphuric acid. With hydrochloric acid present the results are unreliable.

A COMMON method for the separation of silver, in a wide variety of products, consists in its precipitation as silver chloride. Precipitation is usually carried out in the presence of nitric acid or sulphuric acid, in suitable concentration, the precipitate being filtered off and the determination finished gravimetrically or volumetrically.[1]

In the determination of silver in certain residues and complex aluminium alloys, the use of perchloric acid was found to have certain advantages. Furthermore, its use was considered to be necessary at the dissolution stage in the analysis of complex nickel-base alloys. Accordingly, a search of the analytical literature was made, but no reference could be found dealing with the precipitation of silver as silver chloride in perchloric acid solutions. In the development of a suitable method for the determination of up to about 1% silver in the above alloys, therefore, a limited study was made of the effect of perchloric acid on the precipitation of silver chloride under various conditions of acidity.

Experimental Work

The first series of tests covered the precipitation of 4 to 20 mg. of silver as silver chloride in the presence of perchloric acid, the results being compared with those obtained by precipitation in the presence of a small amount of nitric acid only.

The following procedure was used :—

Various aliquots of 0.01N silver nitrate solution were taken to cover the range stated, and these were diluted to just under 200 ml. Varying amounts of perchloric acid (S.G. 1.54), together with two drops of nitric acid (S.G. 1.42), were then added and the solution brought to the boil. To each sample, 2 ml. of approximately 0.2N hydrochloric acid (sufficient to precipitate approximately 0.04 g. of silver) were added to precipitate the silver, and the solutions boiled gently for ten minutes, cooled and allowed to stand in a cool, dark place for 48 hours.

The precipitate was filtered on a 4X sintered glass crucible, using suction, and then washed with cold 3% v/v nitric acid (S.G. 1.42). It was then dissolved into a clean beaker by consecutive treatment with 30 ml. of hot 25% v/v ammonium hydroxide solution (S.G. 0.880), 20 ml. of hot water, 15 ml. of hot 50% v/v nitric acid, 20 ml. hot water, 30 ml. of hot 25% v/v ammonium hydroxide solution and, finally, twice with 20 ml. of hot water. The nitric acid was used to dissolve any metallic silver formed by the action of daylight. The cold filtrate was then made just acid to litmus with 50% v/v nitric acid, thus reprecipitating the silver chloride. 10 ml. of 50% v/v ammonium hydroxide solution and

5 ml. of 10% potassium iodide solution were added and the solution titrated till clear with 0.01N potassium cyanide solution, and then back-titrated with 0.01N silver nitrate solution. A blank solution was processed similarly. The results are recorded in Table I.

TABLE I.

Perchloric acid (S.G. 1.54) % v/v	Silver added g.	Silver found g.	Error g.
0 ⎫	0.0086	0.0084	−0.0002
0 ⎬ Normal	0.0107	0.0107	NIL
0 ⎬ Method	0.0160	0.0156	−0.0004
0 ⎬	0.0173	0.0172	−0.0001
0 ⎭	0.0214	0.0214	NIL
7.5	0.0043	0.0043	NIL
7.5	0.0053	0.0053	NIL
7.5	0.0086	0.0086	NIL
7.5	0.0107	0.0106	−0.0001
7.5	0.0107	0.0107	NIL
7.5	0.0120	0.0118	−0.0002
7.5	0.0214	0.0216	+0.0002
10.0	0.0043	0.0043	NIL
10.0	0.0086	0.0086	NIL
10.0	0.0107	0.0104	−0.0003
10.0	0.0173	0.0173	NIL

It is clear from these results that the precipitation of silver as silver chloride is complete, within the limits of experimental error, in the presence of from 7.5 to 10% v/v perchloric acid (S.G. 1.54), even down to 4–5 mg. quantities.

In the second series of experiments, 5 to 10 mg. of silver were precipitated in the presence of 8.75% v/v perchloric acid with the addition of 3 to 7% v/v nitric acid (S.G. 1.42), 2 to 6% v/v sulphuric acid (S.G. 1.84) and 2 to 5% v/v hydrochloric acid (S.G. 1.18), respectively. The precipitation and final determination was carried out as described above and the results are recorded in Table II.

TABLE II.

Acid Present in addition to 8.75% v/v HClO₄	Acid Concentration % v/v	Silver added g.	Silver found g.	Error g.
Nitric	3	0.0108	0.0107	−0.0001
,,	5	0.0108	0.0108	NIL
,,	5	0.0108	0.0106	−0.0002
,,	7	0.0054	0.0053	−0.0001
,,	7	0.0086	0.0086	NIL
,,	7	0.0108	0.0109	+0.0001
Sulphuric	2	0.0108	0.0110	+0.0002
,,	4	0.0108	0.0107	−0.0001
,,	5	0.0108	0.0106	−0.0002
,,	6	0.0054	0.0054	NIL
,,	6	0.0086	0.0086	NIL
,,	6	0.0108	0.0106	−0.0002
Hydrochloric	2	0.0108	0.0105	−0.0003
,,	4	0.0108	0.0104	−0.0004
,,	5	0.0054	0.0049	−0.0005
,,	5	0.0086	0.0081	−0.0005
,,	5	0.0108	0.0102	−0.0006

1. R. — *Analyst*, 1944, **69**, 270.

Inspection of this table shows that, within the limits of experimental error, complete precipitation of the silver chloride is obtained in the presence of perchloric acid, together with the given percentages of nitric acid and sulphuric acids. In the presence of the hydrochloric acid, however, a significant loss is observed, and as this loss will vary with the temperature of standing, precipitation is considered of little practical value. At 100° C. the silver chloride was completely soluble in the presence of the hydrochloric acid.

It was also observed that, with the smaller amounts of silver, the perchloric acid solutions gave a slightly more coagulative type of precipitate than with the normal method, therefore improving the ease of filtration. With the larger amounts of silver no such improvement was noted.

Conclusions

The above work has shown that, within the limits of the concentrations described, silver may be precipitated quantitatively as silver chloride in the presence of perchloric acid, either alone or together with nitric or sulphuric acid. Low results are obtained in the presence of hydrochloric acid.

Acknowledgment

Useful Devices in the Industrial Chemical Laboratory

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(*Concluded from the July Issue*)

VII. Reducing breakages of measuring flasks.

Measuring flasks of the " sugar flask " type, with wide necks and two calibration marks, which are so very useful for routine purposes, are comparatively "top-heavy " when empty or nearly empty, and the necks break easily when the flasks topple over. This breakage can be entirely prevented by placing around the neck, near the top, a square piece of sheet rubber, about $\frac{1}{8}$ in. to $\frac{1}{4}$ in. thick, of the type used by plumbers, and of a sufficient size to prevent the neck of the flask touching the bench if it topples. This is illustrated in Fig. 7. A hole of a size to give a good fit is made with a wide cork borer and the rubber piece can then be forced over the flared top of the flask neck. The square rubbers which have the advantage of also preventing the flasks from rolling, can be cut with a knife ; if a round shape is preferred, this may be punched out.

Similar devices are obtainable commercially in a few sizes for Nessler tubes or larger measuring cylinders[5] but the home made article is more flexible and can easily be

made to suit various equipment for which the commercial article is unsuitable.

VIII. Support for funnels.

From the same type of rubber sheet can be made funnel supports for filtration into test tubes, measuring and conical flasks, Phillips beakers, etc. Their use prevents breakage or chipping which frequently occurs when large numbers of funnels are hastily placed in the necks of test tubes or flasks. Air locks, which often occur during filtration if the funnel is placed directly into the tube or flask, particularly if wet, are also prevented by their use. Although the development of air locks under the rubber support is unlikely, drilling a small hole close to the hole through which the funnel is inserted, or cutting a shallow radial groove on the lower surface of the rubber will ensure complete freedom from this trouble.

IX. Attaching rubber or glass stoppers to measuring flasks (or cylinders) separating funnels, etc.

Even if the stoppers are interchangeable, it is often desirable to tie the stopper to the neck of the flask, so that it cannot be mislaid or does not have to be held or placed on a clean support whilst the flask is opened. Ordinary string tends to curl up when it gets wet and to rot, particularly if corrosive liquids are used ; it then has a nasty habit of breaking just at the inopportune moment. Waxed thread is slightly better but not really satisfactory. Polyvinyl thread of approximately 1 mm. thickness is practically indestructible, except by organic solvents. It is obtainable at Woolworth's for a few pence under the name of " Plastic thread for 101 uses." It resists immersion in 1:1 sulphuric acid or 8 N caustic soda for at least a week without any sign of deterioration and does not curl up when wet.

X. Air trap for maintaining a CO_2 atmosphere in a conical or similar flask.

For the determination of tin in aluminium alloys, and for other purposes, it is necessary to maintain an inert atmosphere—e.g., of CO_2, in the reaction vessel. This is usually achieved by closing the vessel by a rubber bung fitted with a delivery tube so bent that the lower

5 They are, e.g.—listed in the catalogue of Messrs. Griffin and Tatlock, Kemble Street, London, W.C.2, under the name "Cylinder protectors (LIFE-BUOYS)," Cat. No. 1323-390.
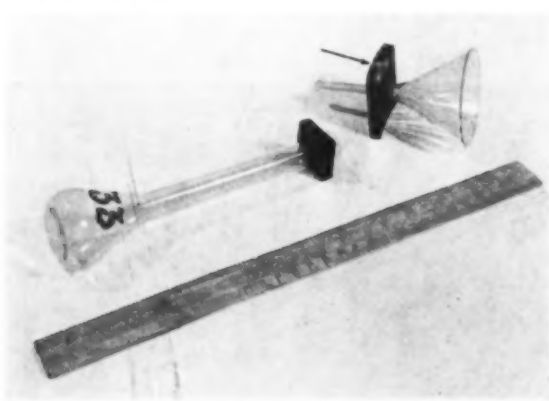

Fig. 7.—Rubber protector for neck of measuring flask and rubber support for funnel, with groove to prevent airlock—indicated by arrow.

end dips into a solution of bicarbonate; the liquid acts as trap during the heating; during the cooling a little of the bicarbonate is sucked back, gives off CO_2 if the reaction mixture is sufficiently acid and thus equalises the pressure.

This is quite satisfactory if the number of determinations is small, but handling of larger numbers is very much easier with the device shown in Fig. 8[6]. It is a modification of the Contat-Göckel valve[7] but is less fragile and much more resistant to thermal shocks.

The dimensions are not critical; an overall height of $5\frac{1}{2}$ in. for the glass bulb has proved satisfactory for 750 ml. conical flasks, without making the flask too top-heavy, even when the bulb is completely filled with the bicarbonate solution. The descending tube a is kept thinner than the ascending part in order to slow down the rate at which liquid is sucked back, otherwise the gas development tends to become too violent.

The flasks thus fitted can be handled with one hand, placed on or removed from the hotplate, immersed in a cooling bath and aligned for titration, all this with the trap in position, with the same ease as ordinary flasks.

XI. Adjustable overflow for cooling baths.

The usual procedure of inserting a holed cork, with or without a piece of glass or other tubing, in the outflow of say a sink, and thus determining the depth of the bath, is often not satisfactory, particularly if the depth has to be varied frequently. The tube must be rather wide, if one wants to be certain that the overflow is able to cope with a rapid inflow, and the adjustment of the hole becomes delicate if the tube is to fit sufficiently tightly and yet not too tightly to slide with ease. For many purposes it was found to be a simple and very effective expedient to place in the outflow hole of the sink a conical rubber Gooch crucible adaptor[8]. If the depth is to be increased, one or more adaptors are inserted in the interior of the first adaptor, this being quite easy due to their conical shape, and in this way the overflow (which is very wide and hardly restricts the outflow hole of the sink) can be built up to a height of several inches.

XII. Reinforced Mohr-Clips.

It has been the experience of many chemists that a good deal of the war and post-war rubber tubing is too hard to be controlled by the commercial Mohr clips. The pressure exerted by these clips can, however, be increased by fitting them with a spring[9] in the manner shown in Fig. 9.

XIII. Plastic Tubing.

The poor quality of much of the post war rubber tubing, as regards resistance to chemical attack and rapidity of perishing has led to its substitution by highly chemical-resistant plastic tubing in many instances. Among the great variety of products obtainable commercially from many sources, the author has found the following three types to be particularly useful.
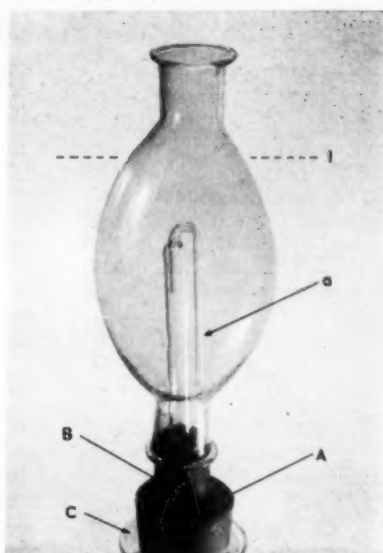
(a) An almost transparent polyvinylchloride (PVC)

A.—Rubber bung, supporting the trap closing the neck C of the conical flask to to which the trap is fitted.
B.—Smaller rubber bung, with the smaller end upwards, closing the lower aperture of the trap.
a.—Narrow descending part of the tube having its aperture about 1 in. below the place indicated by the arrow.
I.—Level to which the trap is filled with saturated bicarbonate solution; in the case of extended boiling time, solid bicarbonate can be added.

Fig. 8.—Air trap, for maintaining CO_2 atmosphere.

tubing[10] which is somewhat harder and more rigid than Type (b) (see below), so that it cannot be controlled by a spring clip. When the end is immersed in boiling water (or rotated cautiously about 6 in. above a Bunsen flame until it gets opaque and starts to emit vapours) it becomes quite soft, and in this condition it can easily be slipped over glass or metal tubing of considerably greater diameter, particularly if a drop of glycerol is used for lubrication. On cooling, the PVC tubing becomes transparent again and contracts somewhat, so that it grips the glass very firmly, particularly if no glycerol is used. If it is intended to connect and disconnect glass and PVC tubing frequently, it is useful to give the end of the plastic tubing a slightly funnel-like shape by heating it over the Bunsen flame, as described above, and forcing into it a conical object lubricated with glycerol. On cooling, the plastic retains the funnel shape which greatly facilitates the insertion of glass or metal tubing.

Teats can easily be made from this material by heating one end over the flame, quickly sealing the edges, either by compression with a strong screw clip, or preferably with a pair of pliers having rather long and

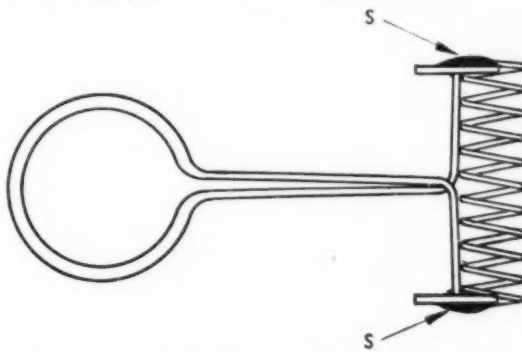10 Obtainable, e.g.—from J. W. Towers, Ltd., Widnes, Lancs.

Fig. 9.—Reinforced Mohr clip (S indicates solder)

6 The author has had no part in developing this piece of apparatus which was in daily use in this laboratory when he joined it. He has been unable to ascertain its origin, but many visitors have found it very useful and stated that they have not seen it before. No description of it was found in the literature nor in the catalogues of laboratory suppliers. The writer thinks, therefore, that some readers may be interested in this description.
7 For a description of the Contat-Göckel valve see e.g., Treadwell and Hall, *Analytical Chemistry*, 9th Edn., 1942, Vol. II, p. 540.
8 e.g. Cat. No. A.2141, Messrs. George & Beckers.
9 Compression spring No. 762, 5/16" x 20 S.W.G. (H. Terry and Sons, Ltd., Redditch) is suitable; this is supplied in 1 ft. lengths, from which a suitable piece may be cut. The two ends are then soldered to the outer part of the plates of the clip. The correct length is best tried out before soldering.

smooth branches. Similarly, "bobbys" can be made and the edges of the seal can be easily trimmed with scissors.

It is also possible to make T-pieces, for example, by the following technique. A hole is drilled in one wall with a thin cork borer. A glass or metal rod, thin enough to slide easily, is then inserted in this piece of tubing to give it support for the next operation. The second piece, to be sealed to the hole in the first piece, is pushed through a suitable glass or, preferably, metal tube (e.g., a cork borer) so that about $\frac{1}{4}$ in. of the plastic protrudes. This protruding end is heated over the flame and then quickly everted over the edge of the glass (or metal) tube. The edge of the hole in the first, and the everted edge of the other piece are then cautiously heated, quickly pressed together and the seal completed with the fingers or tweezers, repeating the heating if necessary.

(b) A softer translucent polyvinylchloride tubing of more rubber-like texture is marketed as surgical dainage tubing, under the trade name " Portex,"[11] in a variety of sizes and qualities. The "elastic" grade is soft enough to be controlled by a reinforced spring clip of the type described above. The "H" grade is slightly tougher and can be used for vacuum work without collapsing. The "standard" type is intermediate. Type (b) can be softened, expanded and sealed just as Type (a); it is slightly more expensive than Type (a) although still less expensive than ordinary rubber tubing, and considerably less expensive than rubber pressure tubing. For some purposes the "Portex" tubing is definitely superior to the type (a) because of its mechanical properties. For other purposes, however, the slightly stiffer texture of Type (a) is an advantage, so that it seems advantageous to stock both varieties in the laboratory. In many cases, various types or sizes can be combined in a useful way, glass or metal connections often being unnecessary.

If a tube can be inserted into the next bigger size with a good fit, the two can be sealed in the following way, if one of the two pieces is reasonably short. Insert from the shorter end a glass or metal rod, just sliding easily into the narrower of the two tubes. After pushing the wider tube over the other to overlap for about $\frac{1}{4}$ in., warm the joint carefully by rotating over a flame until soft and maintain the temperature for a sufficient time to let the heat penetrate to the inner tube ; then apply circular pressure from the outside with a heated tongue-like instrument with suitably curved branches. Such a tool can easily be made from one of the conventional crucible tongues, the nickel ones being very suitable. This all-round pressure on the tubes, stiffened by the inserted rod, produces the weld.

By welding together several short pieces of decreasing diameters, a neat flexible connection between quite large and rather narrow parts of apparatus can be achieved.

(c) Polythene tubing. Most of the specimens of tubing made from this particularly inert material, which the author has been able to see were very thick walled and lacked flexibility. A thin walled and more flexible type is, however, produced for surgical purposes[12] and this should be a valuable addition to the PVC tubing. It

11 Portland Plastics, Ltd., Abey House, 6-10, Victoria Street, London.
12 The firm producing Type (b) of the PVC tubing is also making this type of polythene tubing, mostly in rather small sizes—one with an inner diameter of mm being the largest size,

is slightly stiffer than the PVC tubing and can therefore—using glycerol as a lubricant—be threaded through a hole in a rubber bung or through the lumen of a PVC tubing of Type (a) or (b) with a sufficiently tight fit to be liquid tight. Thus the polythene tubing can, with the help of one or several suitable pieces of PVC tubing, be connected to glass tubing of very much greater diameter and, if the connection is made " glass to polythene " the greater chemical resistance of the polythene is fully utilized.

The author's experience with these plastic tubings has not yet been sufficiently long to make possible a final judgement about the keeping quality of these materials over an extended period of time, but he feels confident that they form a useful addition to the standard equipment of chemical laboratories.

XIV.—Protecting Paper Labels with PVC Sheet

A considerable degree of protection against moisture, corrosive liquids, etc., can be given to paper labels by covering them with a piece (slightly bigger than the label) of transparent thin PVC sheet. It was found best to use Indian ink. The paper label is pasted on the bottle with ordinary paste or glue. When dry, a layer of colourless rubber solution (of the type used for mending bicycle tyres) is spread over the paper and slightly beyond. Another layer is spread over the PVC film. When the bulk of the solvent is evaporated and the surfaces have become tacky, the film is placed in position and firmly pressed down.

The author considers this procedure more satisfactory than various recommended methods, using shellac, ethylcellulose and similar.

Acknowledgment

The described devices were developed in the laboratory of Messrs. International Alloys, Ltd., Aylesbury, and the author wishes to thank the Directors for permission to publish.

Spectrophotometry

RELIABLE spectrophotometric data should now be more easily obtained with the aid of a new booklet, *Spectrophotometry*, recently issued by the National Bureau of Standards and made available from the U.S. Government Printing Office.

The techniques and data resulting from the Bureau's extensive experience in spectrophotometry are presented in this guide so that users of spectrophotometry can better understand their instruments, calibrate and maintain them in the proper operating condition, and guard against the numerous errors common in such work. Instruments and methods for use in the ultra-violet, visible and near-infrared spectral regions are considered, including photographic, visual and photo-electric methods. Important topics covered include definitions of spectrophotometric terms, essential parts of spectrophotometers, typical instruments in current use, types of errors which usually occur in spectrophotometric work, and presentation of standard data for checking the calibration of spectrophotometers. In addition, a bibliography of 127 related references is given.

Circular 484, *Spectrophotometry*, by Kasson S. Gibson (48 large double column pages, illustrated), is available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C., at 25 cents.

First International Microchemistry Congress

An account of the Proceedings at Graz

WHEN one discusses the early history of microchemistry, the names of Friedrich Emich and Fritz Pregl immediately come to mind. Emich can probably be regarded as the true founder of microchemistry. Before he carried out his work there had been, it is true, isolated instances of workers devising methods which nowadays we would describe as microchemical. But Emich, with a broader vision, made a general and a highly successful attack on the whole field of small-scale work.

It was remarkable, and at the same time fortunate, that working in the same city of Graz, Pregl had the need, in the field of elementary organic quantitative analysis, of methods such as those devised by Emich or rendered practicable by his work. Pregl's rapid development of this phase of microchemistry and his ability to interest and instruct others in the methods led to the rapid and world-wide spread of this particular branch of microchemical procedures. Indeed, for a time the part tended to overshadow the whole, so that in many places the term microchemistry was used erroneously but commonly as implying only organic quantitative microanalysis. Latterly this misconception has largely been removed, so that nowadays a truer picture of the scope and nature of microchemistry is more usual. With this comes as a corollary a more proper evaluation of the importance of the work of Emich.

The present year brings us to the twentieth anniversary of the death of Pregl and the tenth anniversary of that of Emich. It was, therefore, highly appropriate that the Austrian Microchemical Society should seize the opportunity to hold in Graz, the city in which Emich and Pregl worked, the first international congress devoted entirely to the subject of microchemistry. That the congress was hopefully regarded by its organisers as the first of many, a hope which is likely to be realised, is indicated by their styling it the "First International Microchemical Congress." Its truly international nature, and the world-wide application of microchemistry to chemical problems are amply illustrated by the fact that about 600 members, representing 29 nations, were enrolled for the Congress. As one would expect, Austrian members represented quite a high proportion of the total membership. Nevertheless, well over 300 of those attending the Congress came from outside Austria. As those familiar with microchemical literature would expect, both Great Britain and America, North and South, were well represented. The only matters for regret on this score were that Feigl, whose name ranks with those of Emich and Pregl as a pioneer in the microchemical field, and Donau, whose collaboration with Emich is well-known, were unable to be present.

The scope of the Congress, which lasted from 2nd to 6th July, can best be indicated by listing the different heads under which the activities seem best to fall, and each of which had its own distinguishing features (1) an exhibition of apparatus and literature : (2) the formal opening meeting of the Congress : (3) the scientific papers presented at the Congress : (4) international discussions on the present and future of microchemistry :

and (5) the social side. Each of these arbitrary divisions deserves brief description and comment.

Exhibition of Equipment

The exhibition of microchemical apparatus and literature, which remained open for the duration of the congress, was representative of all aspects of microchemistry. Manufacturers from Austria, France, Germany, Great Britain, Sweden and Switzerland were well represented. The apparatus on exhibition ranged from a wide selection of microchemical balances, through standard and newly designed apparatus for organic analysis, apparatus for general and microchemical operations, to a fine display of microscopes and accessories for chemical microscopy. On the literature side, visitors were able to inspect display copies of classical microchemical publications of historical interest, side by side with the latest books published in this field in many European languages.

Formal Opening Meeting

On the afternoon of July 2nd a memorial tablet to Emich, set on a wall on the top of the Schlossberg, was unveiled, and this act linked itself naturally with the formal opening of the Congress, which took place on the morning of July 3rd. This opening meeting, which was held in the Stephaniensaal, was under the chairmanship of Professor Lieb, President of the Austrian Microchemical Society and of the Congress. Professor Lieb first welcomed the members, and then received, on behalf of the Austrian Microchemical Society, addresses of of welcome from the major countries participating in the Congress. That from the Microchemistry Group of Great Britain was presented by Mr. R. Belcher of Birmingham University, Chairman of the Group, supported by Mr. D. Phillips, the Hon. Secretary.

Next, the President conferred Honorary Membership of the Austrian Microchemical Society on seven microchemists of world-wide reputation. Of these, five are of Austrian origin, though several are no longer associated with that country : H. K. Alber, of Philadelphia, who was born in Austria, and is well-known for his contributions particularly to organic microchemistry ; A. A. Benedetti-Pichler, born in Vienna, who studied in Vienna and Graz, and now works in New York, where he has made himself recognised as one of the outstanding authorities on various aspects of inorganic microchemistry ; J. Donau, who worked with Emrich in Graz for many years : F. Feigl, the pioneer of drop reactions, formerly of Vienna and now carrying on his researches in Rio de Janeiro ; and R. Strebinger of Vienna, who among other things was responsible for the foundation of *Mikrochemie*, the international journal for microchemistry.

The remaining two honorary members were chosen from France and Great Britain, C. Duval of the Sorbonne, who is particularly known for his contributions to inorganic microanalysis, and R. Belcher, who in his scientific papers and books has contributed to many aspects of the subject, both pure and applied.

A bronze bust of Emich was then unveiled by his daughter, and the meeting closed with appreciations of the work of Pregl, delivered by Professor Lieb, and of Emich, delivered by Dr. Benedetti-Pichler.

Scientific Papers

The programme of scientific papers presented to the Congress included 127 titles. One lecture theatre of the University was devoted to general microchemistry, and this section comprised almost one half of the papers. The remainder, which fell under the heading of applied microchemistry, were delivered simultaneously in another lecture theatre. Reading of the papers and their discussion continued from the afternoon of July 3rd to the afternoon of July 6th.

It would be impossible, in the space available, to give details of these papers, which covered a very wide variety of aspects of microchemistry, both theoretical and practical. Some indication of the range will be obtained from the list of papers read by the British delegates, which is as follows : Dr. G. Hodsman of Messrs. L. Oertling, London—" Developments in Microchemical Balance Design ; " A. K. Al Mahdi and Dr. C. L. Wilson of the Queen's University of Belfast—" The Separation of Metal-Organic Complexes ; " R. Belcher and R. Goulden of Birmingham University—" The Determination of Carbon and Hydrogen in Fluorine-Containing Compounds ; " G. Ingram of Messrs. Courtaulds, Ltd., Maidenhead—" Rapid Micro Combustion Methods for the Determination of Elements in Organic Compounds ; " Dr. R. L. Mitchell of the Macauley Institute for Soil Research, Aberdeen—" The Spectrographic Determination of Trace Elements in Rocks, Minerals and Soils ; " Dr. C. E. Spooner, of the National Coal Board, North West Region—" Microchemical Methods Applied to Industrial Materials."

Summaries of almost all the papers are to be found in the June, 1950 issue of the *Österreichische Chemiker-Zeitung*, pp. 94–118, and it is proposed to publish the papers in full in a forthcoming special issue of the journal *Mikrochemie vereinigt mit Mikrochimica Acta.*

International Discussions

During the course of the Congress informal discussions were held between representatives of those countries in which microchemistry is most highly developed. The purpose of these discussions was to consider a number of points of importance for the proper development of the subject.

The problem of the standardisation of microchemical apparatus and methods was fully discussed, and it was decided that a small international committee should be formed whose duty would be, as a first step, to seek the help of the International Standards Organisation in Geneva in compiling and making available to those countries concerned the work which has already been carried out in this direction. Such work on standardisation is particularly advanced in the United States of America and in Great Britain, and in consequence of this the committee consists of Dr. H. K. Alber (U.S.A.), Mr. R. Belcher (Gt. Britain) and Dr. G. Gorbach (Austria).

Dr. M. Zacherl (Austria) was nominated as the Chairman of a further Committee, the remaining members of which he will himself choose, and which will be recognised by the International Union of Chemistry, its field of work to be international matters of particular importance to Microchemists.

Finally, it was agreed to aim at holding the Second International Microchemical Congress in 1954, the venue to be settled at a later date.

These informal decisions were subsequently approved at the closing meeting of the Congress, by the general body of the members of the Congress.

Social Events

It only remains to mention the social side, which was not the least successful. Two informal social evenings were arranged, to as to give members the fullest opportunity possible to mix and to have informal discussions. Receptions were given to the members by the Burgomaster of Graz and by the Governor of the province of Styria, of which Graz is the capital city. At the latter, an entertainment designed to show something of the folk-culture of Styria was presented. Arrangements were made for tours of Graz and the surrounding country. And finally, it was made possible for members, if they so wished, to attend a concert of the Graz Musical Festival.

Those who attended the Congress will long remember it. The highly successul organisation reflects much credit on the Austrian Microchemical Society, and undoubtedly much of the success was due to the able and energetic work behind the scenes, both before and during Congress, by the organising staff, among whom the name of the organising secretary, Dr. H. Malissa of the Graz Technische Hochschule stands out.

British Balances at Graz

In the exhibition of microchemical apparatus, a number of British microchemical balances were on show. A feature of the display of L. Oertling, Ltd., was their new microchemical balance which, in addition to being aperiodic and having weight-loading, provides a direct reading of 1·0 microgram per division of a large illuminated scale. The capacity is 20 g., with a sensitivity of 0·001 mg. per division, the graticule being divided
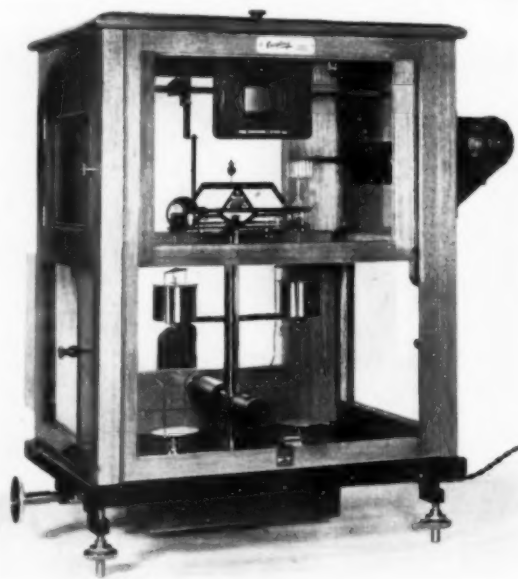
New Oertling aperiodic Microgram balance

100–0–100 and having, therefore, a range of ± 0·1 mg. Made of non-magnetic nickel-chromium alloy, the 5 in. beam has a rider bar serrated into 100 parts for use with a 5 mg. rider. The rider-slide is of the spring loaded vertical type and traverses the whole length of the rider bar. All knife edges and planes are of agate, the centre plane being single-piece. Flat, circular, 1½ in. between wire, pans, fitted with hooks, have adjustable pan supports incorporating special spring-plungers to avoid oscillation when setting the balance in motion by the side-action release which lowers the fulcrum on to the centre plane.

The optical system incorporates a 12 v. projection lamp which, with a corrected condenser system and projection lens gives a very clearly illuminated enlarged graticule range on the large adjustable ground-glass screen in the balance case.

The mechanical fraction-loader, fitted to the right-hand side of the case, consists of a rotating dial which can be turned in either direction to operate a series of levers controlling the addition to and removal from the right arm of the beam of weights up to a total of 0·09 g.

Stanton Instruments, Ltd., displayed two micro-balances, models M.C.1 and M.C.1/A, both of which were introduced about two years ago, but over this time they have been considerably improved. An improved projection screen is now being fitted on both these instruments, also a projection lamp which has very fine adjustment in three directions to permit the maximum amount of light to be obtained from a small wattage bulb. Both instruments are fitted with synthetic sapphire (corrundum) planes.

These micro-chemical, aperiodic, projected reading balances have a capacity of 20 g. and a sensitivity of 0·01 mg. per division, the beam, which is of special light weight construction in hard rolled duralumin, is 10 cms. long with 100 accurately notched divisions. Finest selected agate is used to form the knife edges and as already mentioned the planes are of synthetic sapphire, which experience has shown will maintain the sensitivity two or three times longer than an instrument fitted with agate planes.

Stanton aperiodic Micro-Balance Model M.C.1/A

The optical system comprises a 12 v. 6 W. lamp which is fitted to the rear of the case and, by means of a condenser system and lens, a graticule image is projected on to a ground glass screen situated at an ideal position at the base of the centre pillar. Reading can be taken over a range of ± 1 mg. The graticule is graduated 100 divisions either side of a centre zero, each division representing 0·01 mg.

Vacuum-Fusion Apparatus

IMPURITIES in metals can be detected even when the percentage of a given element is as small as 10^{-5} (0·00001) per cent. or one part in a million; with the vacuum-fusion apparatus in use at the National Research Corporation, Cambridge, Mass. The equipment was designed and assembled by the company's research division as a tool for use in analysing for minute impurities contained in highly purified metals. Ordinary analytical procedures for the determination of the elements in question are impracticable in the percentage range encountered.

The principle is as follows: Under proper melting conditions, high-vacuum metals may be made to give up their gases quantitatively. The combined oxides react with the graphite crucible in which the melting is conducted, and the oxygen is released in the form of carbon monoxide. The hydrides and nitrides are decomposed, and the hydrogen and nitrogen liberated directly. The choice of a correct temperature is highly important. It must be sufficiently high to decompose the chemical compounds and at the same time not so high that the metal being analysed will volatilise and coat the inside of the apparatus. Sometimes it is necessary to add a flux to assist in achieving these results. The mixture of hydrogen, nitrogen, and carbon monoxide is pumped by means of a mercury-diffusion pump into a vacuum chamber of known volume. The pressure of gas collected is measured by a McLeod gauge which is a device intended to measure low gas pressures.

Since the total pressure is low, Dalton's law holds. The total pressure P_T of the gases present is equal to the sum of the pressures which each gas would exert if it alone were present in the given volume.

After the total pressure of the gases is determined they are circulated through a tube containing copper oxide. Here the hydrogen and carbon monoxide take oxygen from the copper oxide and are converted to water vapour and carbon dioxide, respectively. All the gases are allowed to circulate also through a cold trap cooled to −196° C. by liquid nitrogen. Here the carbon dioxide and water vapour are frozen out while the nitrogen remains gaseous. The nitrogen is then returned to the McLeod gauge where its pressure is determined.

The cold trap is then warmed up to about −80° C. with a mixture of dry ice and acetone. At this temperature the CO_2 is set free and its pressure plus the pressure of the nitrogen is measured. Knowing the pressure of the nitrogen and the pressure of the gases, all the necessary data for computation of the weight of each gas present can be obtained. Since the volume in which the gas is compressed is known, the number of molecules of each gas or the weight, can be estimated.

The apparatus itself is constructed entirely of glass and the few ground-glass joints present are carefully

sealed with a special high-vacuum wax having an exceedingly low vapour pressure. Since the pressure in the system should be in the order of 0·01 micron, the smallest leak will vitiate the results.

The main system is evacuated by the combination of a diffusion pump and a high-efficiency mechanical pump. The gases are circulated through the system by means of two small high-speed diffusion pumps.

Since it would be impracticable to place a heater inside a high-vacuum system, the metals are heated from outside the system by means of a high-frequency induction coil.

The time for a single analysis exclusive of the preparation of the sample is about an hour and a half. Accuracy depends a good deal on the individual system and the operator, but with care determinations may be made with a high degree of reproducibility. The quantities detectable may be as low as 10^{-5} per cent., as is the case with hydrogen in many metals.

Correspondence

PERCHLORIC ACID : IS IT POISONOUS?
The Editor, METALLURGIA.
Sir,

The article by Mr. Croall on perchloric acid interested us because we have for a long time advocated the use of this very useful acid and, over many years' experience, we have never encountered any hazards from explosion. We are, however, rather puzzled at the statement that perchloric acid is not poisonous and perhaps Mr. Croall would be good enough to define his conception of poison. I imagine that this acid is equally as poisonous as hydrochloric, nitric or sulphuric acids and all of these have to be sold in bottles labelled " poison." Perchloric acid is not on the Home Office Schedule of Poisons, but we have always assumed it to be poisonous and any information Mr. Croall has on this point would certainly be of interest to us.

Yours faithfully,
G. H. OSBORN
The British Drug Houses, Ltd., *Chief Analyst.*
B.D.H. Laboratory Chemicals Group,
Poole, Dorset.
August 1st, 1950.

The Editor, METALLURGIA.
Sir,

In reply to Mr. Osborn's letter, I would point out that H. H. Willard states in the *J. Am. Chem. Society*, 34, 1480 (1912), that perchloric acid is not poisonous. Lundell, Hoffman and Bright in " Chemical Analysis of Iron and Steel " and Hillebrand and Lundell in " Applied Inorganic Analysis " quote Willard's statement and appear to accept it as true.

Personally, I think that Mr. Osborn correctly infers that a definition is required of poison. The *Encyclopædia Britannica* states " a poison is a substance which, by its direct action on the mucous membrane tissues or skin or after absorption into the circulatory system, can, in the way in which it is administered, injuriously affect health or destroy life." According to this definition, any substance which acts corrosively on the body is a poison. Perchloric acid does act in this way. A small amount of 70% perchloric acid will in a relatively short time produce a faint brown stain on the fingers. There-

fore perchloric acid like other mineral acids should be classed as a corrosive poison.

However, it is true that this action of mineral acids is often thought of as a burning and not as a poisonous effect. Moreover, on dilution these substances lose their poisonous effect, and can then be classed as non-poisonous ; i.e., Dilute hydrochloric acid is used as a medicine.

Although I am not sure, I think that Willard is claiming that if perchloric acid is suitably diluted to remove the burning or corrosive effect due to its acidity then it can be classed as non-poisonous. In other words it is not an irritant poison like arsenic or a systemic poison like opium or alcohol.

Yours sincerely,
G. CROALL,
Senior Research Officer.
G.K.N. Group Services Limited,
G.K.N. Research Laboratories,
Manor Works, Ettingshall,
Wolverhampton.
August 10th, 1950.

THE USE OF GELATINE IN DETERMINING BISMUTH CONTENTS BY ELECTROLYTIC METHODS
The Editor, METALLURGIA.
Sir,

I have read with some interest the article by Mr. Goldberg on the determination of bismuth by electrolytic methods. As Mr. Goldberg points out, the method is not new and most of the principles were laid down about 20 years ago.

We determine the bismuth content of all our bismuth compounds by the direct electrolysis of the bismuth. However, what we have found to be necessary in order to obtain a really good adherent deposit is the addition of a water solution containing 0·2 of a gramme of gelatine. Without this gelatine we frequently found that the bismuth had a tendency to form spongy deposits which could not be removed without loss.

Yours faithfully,
G. H. OSBORN,
The British Drug Houses Ltd., *Chief Analyst.*
B.D.H. Laboratory Chemicals Group,
Poole, Dorset.
August 1st, 1950.

The Editor, METALLURGIA.
Sir,

Thank you for sending along to me the letter of Mr. Osborn of British Drug Houses Ltd., anent my article in METALLURGIA (July, 1950).

After sending the aforementioned article to you for consideration we found two instances where the electrolytic deposit of bismuth was not as adherent as desired. A 1% solution of gum arabia served to remedy that defect and that procedure has been used here since then.

Of course gelatine will serve as well as gum arabia and I wish to thank Mr. Osborn for calling my attention to his use of that reagent.

Sincerely yours,
C. GOLDBERG.
New England Smelting Works, Inc.
W. Springfield,
Mass., U.S.A.
August 7th, 1950.